

Jan Delaval.

Access DB# 121537

SEARCH REQUEST FORM

Scientific and Technical Information Center

Requester's Full Name: Sabika Aziz Examiner #: 74141 Date: 5/7/04
App Unit: 1616 Phone Number: 202-512-2222 Serial Number: 09/335022
Box and Bldg Room Location: 4C70 Room 4A45 Results Format Preferred (circle) PAPER DISK E-MAIL

If more than one search is submitted, please prioritize searches in order of need. MEJ

Please provide a detailed statement of the search topic, and describe as specifically as possible the subject matter to be searched. Include the elected species or structures, keywords, synonyms, acronyms, and registry numbers, and combine with the concept or utility of the invention. Define any terms that may have a special meaning. Give examples or relevant citations, authors, etc., if known. Please attach a copy of the cover sheet, pertinent claims, and abstract.

Title of Invention: Process for Producing Vit. D₃ + Pre-Vit. D₃
Inventors (please provide full names): Monika Johansson

Earliest Priority Filing Date: 6/17/1999

For Sequence Searches Only Please include all pertinent information (parent, child, divisional, or issued patent numbers) along with the appropriate serial number.

Please search for the process of
isolation of Vit. D₃ or pre-Vit. D₃ or steroid
by, as in ch 1-6

Please note
1) normal phase column] can be reversed
Chromatography Extract
before get Col. Chrom.
Straight phase.

2) back-pressure regulation] flash Chromatography

NPL (J. Chromatography?)
Please use Vit. D + separation or isolation for
Text Search

STAFF USE ONLY

Search #	Type of Search	Vendors and cost where applicable
<u>1</u>	NA Sequence (#)	STN <input checked="" type="checkbox"/>
Searcher's Phone # <u>22504</u>	AA Sequence (#)	Dialog
Searcher's Location	Structure (#) <input checked="" type="checkbox"/>	Questel/Orbit
Date Searcher Picked Up <u>5/8</u>	Bibliographic <input checked="" type="checkbox"/>	Dr. Link
Date Completed <u>5/8</u>	Linkage	Lexis/Nexis
Searcher Prep. & Review Time	Fulltext	Sequence Systems
Client Prep. Time <u>1.5</u>	Patent Family	WWW/Internet
<u>44x</u>	Other	Other (specify)

4

=> fil reg

FILE 'REGISTRY' ENTERED AT 15:17:03 ON 08 MAY 2004

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PLEASE SEE "HELP USAGETERMS" FOR DETAILS.

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Property values tagged with IC are from the ZIC/VINITI data file provided by InfoChem.

STRUCTURE FILE UPDATES: 7 MAY 2004 HIGHEST RN 680859-76-1

DICTIONARY FILE UPDATES: 7 MAY 2004 HIGHEST RN 680859-76-1

TSCA INFORMATION NOW CURRENT THROUGH JANUARY 6, 2004

Please note that search-term pricing does apply when conducting SmartSELECT searches.

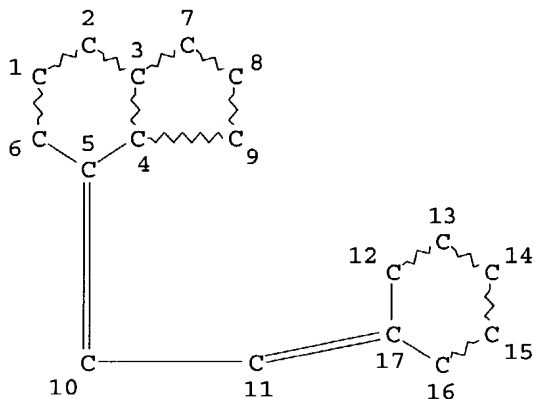
Crossover limits have been increased. See HELP CROSSOVER for details.

Experimental and calculated property data are now available. For more information enter HELP PROP at an arrow prompt in the file or refer to the file summary sheet on the web at:

<http://www.cas.org/ONLINE/DBSS/registryss.html>

=> d sta que 16

L4 STR



NODE ATTRIBUTES:

DEFAULT MLEVEL IS ATOM

DEFAULT ECLEVEL IS LIMITED

GRAPH ATTRIBUTES:

RING(S) ARE ISOLATED OR EMBEDDED

NUMBER OF NODES IS 17

STEREO ATTRIBUTES: NONE

L6 8414 SEA FILE=REGISTRY SSS FUL L4

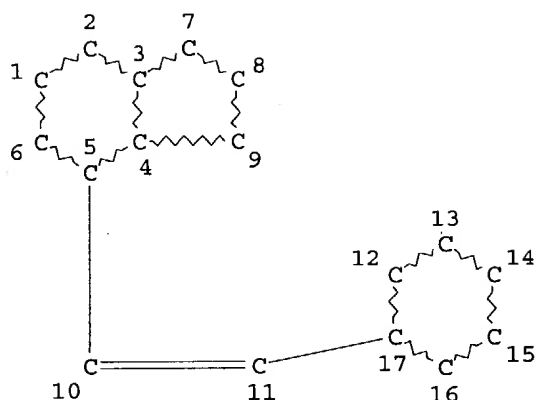
100.0% PROCESSED 15155 ITERATIONS

SEARCH TIME: 00.00.01

8414 ANSWERS

=> => d sta que 19

L7 STR



NODE ATTRIBUTES:
 DEFAULT MLEVEL IS ATOM
 DEFAULT ECLEVEL IS LIMITED

GRAPH ATTRIBUTES:
 RING(S) ARE ISOLATED OR EMBEDDED
 NUMBER OF NODES IS 17

STEREO ATTRIBUTES: NONE
 L9 662 SEA FILE=REGISTRY SSS FUL L7

100.0% PROCESSED 203796 ITERATIONS
 SEARCH TIME: 00.00.02

662 ANSWERS

=> d ide can l1

L1 ANSWER 1 OF 1 REGISTRY COPYRIGHT 2004 ACS on STN
 RN 67-97-0 REGISTRY
 CN 9,10-Secocholesta-5,7,10(19)-trien-3-ol, (3 β ,5Z,7E) - (9CI) (CA INDEX NAME)

OTHER CA INDEX NAMES:

CN Cholecalciferol (8CI)

OTHER NAMES:

CN 9,10-Secocholesta-5,7,10(19)-trien-3 β -ol

CN Arachitol

CN Calciol

CN Colecalciferol

CN D3-Vigantol

CN Delsterol

CN Deparal

CN Devaron

CN FeraCol

CN Granuvit D3

CN NSC 375571

CN Oleovitamin D3

CN Quintox

CN Ricketon

CN Trivitan

CN Vi-De3

CN Videkhol

CN Vigorsan

CN **Vitamin D3**

CN Vitinc Dan-Dee-3

FS STEREOSEARCH

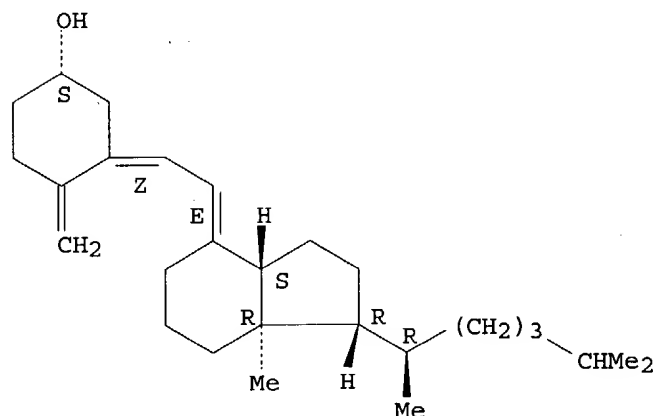
DR 8024-19-9, 8050-67-7

MF C27 H44 O

CI COM

LC STN Files: ADISNEWS, AGRICOLA, BEILSTEIN*, BIOBUSINESS, BIOSIS,
 BIOTECHNO, CABA, CANCERLIT, CAOLD, CAPLUS, CHEMCATS, CHEMLIST, CIN,
 DDFU, DRUGU, EMBASE, HODOC*, HSDB*, IFICDB, IFIPAT, IFIUDB, IPA, MRCK*,
 NIOSHTIC, PS, SPECINFO, TOXCENTER, USAN, USPAT2, USPATFULL, VETU
 (*File contains numerically searchable property data)
 Other Sources: EINECS**, TSCA**
 (**Enter CHEMLIST File for up-to-date regulatory information)

Absolute stereochemistry.
 Double bond geometry as shown.



PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

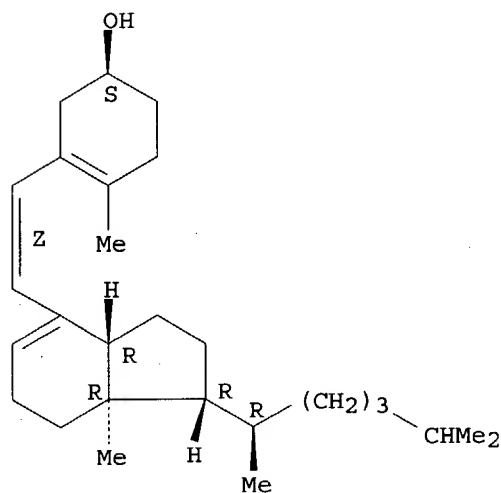
5260 REFERENCES IN FILE CA (1907 TO DATE)
 492 REFERENCES TO NON-SPECIFIC DERIVATIVES IN FILE CA
 5265 REFERENCES IN FILE CAPLUS (1907 TO DATE)
 5 REFERENCES IN FILE CAOLD (PRIOR TO 1967)

REFERENCE 1: 140:326651
 REFERENCE 2: 140:320239
 REFERENCE 3: 140:315647
 REFERENCE 4: 140:309487
 REFERENCE 5: 140:309381
 REFERENCE 6: 140:309014
 REFERENCE 7: 140:302745
 REFERENCE 8: 140:302652
 REFERENCE 9: 140:302466
 REFERENCE 10: 140:300743

=> d ide can l2

RN 1173-13-3 REGISTRY
 CN 9,10-Secocholesta-5(10),6,8-trien-3-ol, (3 β ,6Z)- (9CI) (CA INDEX NAME)
 OTHER CA INDEX NAMES:
 CN 9,10-Secocholesta-5(10),6,8-trien-3 β -ol (8CI)
 CN Precalciferol (6CI)
 CN **Previtamin D3** (7CI)
 OTHER NAMES:
 CN Precalciferol3
 CN Precholecalciferol
 CN Previtamin D
 FS STEREOSEARCH
 MF C27 H44 O
 LC STN Files: AGRICOLA, BEILSTEIN*, BIOBUSINESS, BIOSIS, CA, CAOLD, CAPLUS, CASREACT, CHEMINFORMRX, CHEMLIST, DDFU, DRUGU, EMBASE, IFICDB, IFIPAT, IFIUDB, MEDLINE, PROMT, PS, TOXCENTER, USPATFULL
 (*File contains numerically searchable property data)
 Other Sources: EINECS**
 (**Enter CHEMLIST File for up-to-date regulatory information)

Absolute stereochemistry.
 Double bond geometry as shown.



PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

233 REFERENCES IN FILE CA (1907 TO DATE)
 9 REFERENCES TO NON-SPECIFIC DERIVATIVES IN FILE CA
 234 REFERENCES IN FILE CAPLUS (1907 TO DATE)
 12 REFERENCES IN FILE CAOLD (PRIOR TO 1967)

REFERENCE 1: 140:301671
 REFERENCE 2: 140:162982
 REFERENCE 3: 140:16105
 REFERENCE 4: 139:307389
 REFERENCE 5: 138:255392

REFERENCE 6: 138:234042
REFERENCE 7: 138:136053
REFERENCE 8: 138:102954
REFERENCE 9: 137:302012
REFERENCE 10: 137:67030

=> d ide can l3

L3 ANSWER 1 OF 1 REGISTRY COPYRIGHT 2004 ACS on STN
RN 1406-16-2 REGISTRY
CN **Vitamin D (8CI, 9CI)** (CA INDEX NAME)
MF Unspecified
CI COM, MAN
LC STN Files: ADISNEWS, AGRICOLA, ANABSTR, BIOBUSINESS, BIOSIS, BIOTECHNO,
CA, CABA, CANCERLIT, CAPLUS, CASREACT, CBNB, CEN, CHEMLIST, CIN, CSNB,
DDFU, DIOGENES, DRUGU, EMBASE, IFICDB, IFIPAT, IFIUDB, IPA, MEDLINE,
NAPRALERT, NIOSHTIC, PIRA, PROMT, RTECS*, TOXCENTER, USPAT2, USPATFULL,
VETU
(*File contains numerically searchable property data)
Other Sources: EINECS**, NDSL**, TSCA**
(**Enter CHEMLIST File for up-to-date regulatory information)

*** STRUCTURE DIAGRAM IS NOT AVAILABLE ***

10574 REFERENCES IN FILE CA (1907 TO DATE)

833 REFERENCES TO NON-SPECIFIC DERIVATIVES IN FILE CA

10585 REFERENCES IN FILE CAPLUS (1907 TO DATE)

REFERENCE 1: 140:327125
REFERENCE 2: 140:327062
REFERENCE 3: 140:326651
REFERENCE 4: 140:320585
REFERENCE 5: 140:320583
REFERENCE 6: 140:320475
REFERENCE 7: 140:320433
REFERENCE 8: 140:320428
REFERENCE 9: 140:320350
REFERENCE 10: 140:320336

=> => d ide can l14

L14 ANSWER 1 OF 1 REGISTRY COPYRIGHT 2004 ACS on STN
RN 124-38-9 REGISTRY
CN **Carbon dioxide (8CI, 9CI)** (CA INDEX NAME)
OTHER NAMES:
CN Carbon oxide (CO2)
CN Carbon-12 dioxide
CN Carbon-12C dioxide-16O2

CN Carbonic acid anhydride
CN Carbonic acid gas
CN Carbonic anhydride
CN Dry ice
CN Khladon 744
CN R 744
FS 3D CONCORD
DR 18923-20-1
MF C 02
CI COM
LC STN Files: ANABSTR, BIOSIS, CA, CAOLD, CASREACT, CHEMCATS, CHEMLIST,
CIN, CSCHM, CSNB, DIPPR*, GMELIN*, HODOC*, IFICDB, IFIPAT, IFIUDB,
MEDLINE, PDLCOM*, RTECS*, SPECINFO, TOXCENTER, USAN, USPAT2, USPATFULL,
VTB
(*File contains numerically searchable property data)
Other Sources: DSL**, EINECS**, TSCA**
(**Enter CHEMLIST File for up-to-date regulatory information)

O=C=O

PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

176638 REFERENCES IN FILE CA (1907 TO DATE)
678 REFERENCES TO NON-SPECIFIC DERIVATIVES IN FILE CA
176812 REFERENCES IN FILE CAPLUS (1907 TO DATE)
21 REFERENCES IN FILE CAOLD (PRIOR TO 1967)

REFERENCE 1: 140:331428
REFERENCE 2: 140:331427
REFERENCE 3: 140:330835
REFERENCE 4: 140:330508
REFERENCE 5: 140:330413
REFERENCE 6: 140:329684
REFERENCE 7: 140:329637
REFERENCE 8: 140:329394
REFERENCE 9: 140:329119
REFERENCE 10: 140:328988

=> d his

(FILE 'HOME' ENTERED AT 14:46:34 ON 08 MAY 2004)
SET COST OFF

FILE 'REGISTRY' ENTERED AT 14:46:51 ON 08 MAY 2004
E VITAMIN D3/CN

L1 1 S E3
E PREVITAMIN D3/CN
L2 1 S E3
E VITAMIN D/CN
L3 1 S E3

L4 STR
 L5 50 S L4
 L6 8414 S L4 FUL
 SAV TEMP L6 QAZI335/A
 L7 STR L4
 L8 0 S L7
 L9 662 S L7 FUL
 SAV L9 TEMP QAZI335A/A
 L10 9076 S L6,L9
 L11 9074 S L10 NOT L1,L2
 L12 366 S L11 AND ?VITAMIN?/CNS
 L13 8708 S L11 NOT L12
 L14 1 S CARBON DIOXIDE/CN

FILE 'HCAPLUS' ENTERED AT 14:52:16 ON 08 MAY 2004

L15 5267 S L1
 L16 235 S L2
 L17 12688 S (VIT OR VITAMIN) (L) D3
 L18 136 S (VIT OR VITAMIN) (L) D 3
 L19 2247 S CHOLECALCIFEROL#
 L20 14 S COLECALCIFEROL#
 L21 249 S (PREVIT OR PREVITAMIN OR PRE() (VIT OR VITAMIN)) (L) D3
 L22 -6 S (PREVIT OR PREVITAMIN OR PRE() (VIT OR VITAMIN)) (L) D 3
 L23 83 S PRECALCIFEROL# OR PRE CALCIFEROL#
 L24 125 S PREVITAMIN D
 L25 10602 S L3
 L26 29557 S VITAMIN D#
 L27 16535 S L12
 L28 3123 S L13
 L29 36305 S L15-L28
 E JOHANNSEN M/AU
 L30 17 S E3,E8
 L31 2 S L30 AND L29
 L32 270 S L29 AND (LAROCHE? OR LA ROCHE? OR HOFFMANN?)/PA,CS
 L33 1 S US20010001801/PN OR EP98-111490/AP,PRN
 L34 176984 S L14
 L35 441586 S CARBON DIOXIDE OR CO2
 L36 204 S CARBONDIOXIDE
 L37 184 S L29 AND L34-L36
 L38 4 S L37 AND L31,L32,L33
 E CHROMATOGRAPHY/CT
 L39 1 S L37 AND E30
 L40 3 S L37 AND E110
 L41 2 S L37 AND E145,E151,E154
 L42 0 S L37 AND E158
 L43 2 S L37 AND E27-E29
 E E3+ALL
 L44 2 S L37 AND E4-E6
 L45 12 S L37 AND E3+NT
 E SILICA GEL/CT
 L46 2 S L37 AND E4-E28
 E E3+ALL
 L47 2 S L37 AND E16,E15+NT
 L48 18 S L37 AND (?SUPERCRIT? OR ?SUPER CRIT?)
 L49 1 S L37 AND FLASH?
 L50 23 S L33,L38-L49
 E SEPARATION/CT
 L51 561 S E3+OLD,NT,PFT AND L29
 E PURIFICATION/CT
 L52 9 S E3+OLD,NT,PFT AND L29
 E ISOLATION/CT
 L53 561 S L51-L52
 L54 19 S L37 AND (?SUPERCRIT? OR ?SUPER CRIT? OR FLASH?)

L55 0 S L37 AND (BACKPRESSUR? OR BACK PRESSUR?)
L56 22 S L37 AND ?CHROMATOG?
L57 30 S L54,L56,L50
L58 20 S L57 AND (PY<=1999 OR PRY<=1999 OR AY<=1999)
L59 473 S (PROVIT? OR PRO VIT?) ()D#
L60 172 S L59 NOT L29
L61 163 S L60 AND (PY<=1999 OR PRY<=1999 OR AY<=1999)
L62 23 S L61 AND ?CHROMATO?
L63 0 S L61 AND (BACKPRESSUR? OR BACK PRESSUR?)
L64 0 S L61 AND (?SUPERCRIT? OR ?SUPER CRIT? OR FLASH?)
L65 7 S L61 AND L34-L36
L66 2 S L61 AND CHROMATOGRAPHY+OLD,NT,PFT/CT
L67 2 S L61 AND SEPARATION+OLD,NT,PFT/CT
L68 0 S L61 AND PURIFICATION+OLD,NT,PFT/CT
L69 49 S L62,L65-L67,L58
L70 49 S L69 AND (PY<=1999 OR PRY<=1999 OR AY<=1999)
L71 10 S L57 NOT L70
L72 8 S L70 AND ?CRITIC?
L73 27 S L70 AND L34-L36
L74 27 S L72,L73
L75 22 S L70 NOT L74
SEL DN AN L74 25
L76 26 S L74 NOT E1-E3
L77 36 S L71,L76
L78 12 S L77 AND (?RADIAT? OR UV OR ULTRAVIOL? OR ULTRA VIOL?)
L79 36 S L77,L78

FILE 'REGISTRY' ENTERED AT 15:17:03 ON 08 MAY 2004

=> fil hcaplus

FILE 'HCAPLUS' ENTERED AT 15:18:23 ON 08 MAY 2004

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FILE COVERS 1907 - 8 May 2004 VOL 140 ISS 20

FILE LAST UPDATED: 7 May 2004 (20040507/ED)

This file contains CAS Registry Numbers for easy and accurate substance identification.

=> d 179 all hitstr tot

L79 ANSWER 1 OF 36 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 2003:855901 HCAPLUS

DN 139:354553

ED Entered STN: 31 Oct 2003

TI Extraction of lipophilic compounds for cosmetic and pharmaceutical uses

IN Catchpole, Owen John; MacKenzie, Andrew Douglas; Grey, John Bertram

PA Industrial Research Limited, N. Z.

SO PCT Int. Appl., 49 pp.

CODEN: PIXXD2

DT Patent

LA English

IC ICM C07C067-56

ICS C11B007-00; C11C001-08

CC 63-8 (Pharmaceuticals)

Section cross-reference(s): 9, 17, 62

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	WO 2003089399	A1	20031030	WO 2003-NZ62	20030411
	W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG				

PRAI NZ 2002-518504 A 20020422

AB A process for extracting a wide range of lipophilic compds. from urea-containing

solns. is described. The process utilizes a near-critical fluid as the extraction

solvent. The process is particularly applicable to the extraction of polyunsatd. fatty acids from the filtrate obtained upon urea fractionation, as employed in the processing of fish and other oils. In contrast to known processes, the lipophilic compds. may be extracted without the use of non-food grade solvents, and are suitable for pharmaceutical and cosmetic uses. Thus, free fatty acids (FFA) were recovered from fish oil glycerides followed by urea complexation. The polyunsatd. fatty acids content was 40.8%. The total recovery of FFA was 86%.

ST extn lipophilic compd cosmetic pharmaceutical

IT Fats and Glyceridic oils, biological studies

RL: COS (Cosmetic use); PEP (Physical, engineering or chemical process); PYP (Physical process); THU (Therapeutic use); BIOL (Biological study); PROC (Process); USES (Uses)

(Biota orientalis; extraction of lipophilic compds. for cosmetic and pharmaceutical uses)

IT Fats and Glyceridic oils, biological studies

RL: COS (Cosmetic use); PEP (Physical, engineering or chemical process); PYP (Physical process); THU (Therapeutic use); BIOL (Biological study); PROC (Process); USES (Uses)

(Currant (Ribes nigrum); extraction of lipophilic compds. for cosmetic and pharmaceutical uses)

IT Alcohols, biological studies

RL: COS (Cosmetic use); PEP (Physical, engineering or chemical process); PYP (Physical process); THU (Therapeutic use); BIOL (Biological study); PROC (Process); USES (Uses)

(C1-4; extraction of lipophilic compds. for cosmetic and pharmaceutical uses)

IT Fats and Glyceridic oils, biological studies

RL: COS (Cosmetic use); PEP (Physical, engineering or chemical process); PYP (Physical process); THU (Therapeutic use); BIOL (Biological study); PROC (Process); USES (Uses)

(Echium; extraction of lipophilic compds. for cosmetic and pharmaceutical uses)

IT Fats and Glyceridic oils, biological studies

RL: COS (Cosmetic use); PEP (Physical, engineering or chemical process); PYP (Physical process); THU (Therapeutic use); BIOL (Biological study);

- PROC (Process); USES (Uses)
(*Limnanthes alba* seed; extraction of lipophilic compds. for cosmetic and pharmaceutical uses)
- IT Fats and Glyceridic oils, biological studies
RL: COS (Cosmetic use); PEP (Physical, engineering or chemical process);
PYP (Physical process); THU (Therapeutic use); BIOL (Biological study);
PROC (Process); USES (Uses)
(*Lunarium*; extraction of lipophilic compds. for cosmetic and pharmaceutical uses)
- IT Fats and Glyceridic oils, biological studies
RL: COS (Cosmetic use); PEP (Physical, engineering or chemical process);
PYP (Physical process); THU (Therapeutic use); BIOL (Biological study);
PROC (Process); USES (Uses)
(animal, hydrolyzates; extraction of lipophilic compds. for cosmetic and pharmaceutical uses)
- IT Fats and Glyceridic oils, biological studies
RL: COS (Cosmetic use); PEP (Physical, engineering or chemical process);
PYP (Physical process); THU (Therapeutic use); BIOL (Biological study);
PROC (Process); USES (Uses)
(borage seed; extraction of lipophilic compds. for cosmetic and pharmaceutical uses)
- IT Fatty acids, biological studies
RL: COS (Cosmetic use); PEP (Physical, engineering or chemical process);
PYP (Physical process); THU (Therapeutic use); BIOL (Biological study);
PROC (Process); USES (Uses)
(esters, with C1-4 alcs.; extraction of lipophilic compds. for cosmetic and pharmaceutical uses)
- IT Fats and Glyceridic oils, biological studies
RL: COS (Cosmetic use); PEP (Physical, engineering or chemical process);
PYP (Physical process); THU (Therapeutic use); BIOL (Biological study);
PROC (Process); USES (Uses)
(evening primrose; extraction of lipophilic compds. for cosmetic and pharmaceutical uses)
- IT Extraction
(extraction of lipophilic compds. for cosmetic and pharmaceutical uses)
- IT Carotenes, biological studies
Fatty acids, biological studies
Glycerides, biological studies
Hydrocarbons, biological studies
Sterols
Vitamins
Waxes
RL: COS (Cosmetic use); PEP (Physical, engineering or chemical process);
PYP (Physical process); THU (Therapeutic use); BIOL (Biological study);
PROC (Process); USES (Uses)
(extraction of lipophilic compds. for cosmetic and pharmaceutical uses)
- IT Vitamins
RL: COS (Cosmetic use); PEP (Physical, engineering or chemical process);
PYP (Physical process); THU (Therapeutic use); BIOL (Biological study);
PROC (Process); USES (Uses)
(fat-soluble; extraction of lipophilic compds. for cosmetic and pharmaceutical uses)
- IT Alcohols, biological studies
Amides, biological studies
RL: COS (Cosmetic use); PEP (Physical, engineering or chemical process);
PYP (Physical process); THU (Therapeutic use); BIOL (Biological study);
PROC (Process); USES (Uses)
(fatty; extraction of lipophilic compds. for cosmetic and pharmaceutical uses)
- IT Fats and Glyceridic oils, biological studies
RL: COS (Cosmetic use); PEP (Physical, engineering or chemical process);
PYP (Physical process); THU (Therapeutic use); BIOL (Biological study);

- PROC (Process); USES (Uses)
(fish, hydrolyzates; extraction of lipophilic compds. for cosmetic and pharmaceutical uses)
- IT Hydrocarbons, processes
RL: PEP (Physical, engineering or chemical process); PYP (Physical process); PROC (Process)
(fluoro; extraction of lipophilic compds. for cosmetic and pharmaceutical uses)
- IT Lipids, biological studies
RL: COS (Cosmetic use); PEP (Physical, engineering or chemical process); PYP (Physical process); THU (Therapeutic use); BIOL (Biological study); PROC (Process); USES (Uses)
(hydrolyzates; extraction of lipophilic compds. for cosmetic and pharmaceutical uses)
- IT Fatty acids, biological studies
RL: COS (Cosmetic use); PEP (Physical, engineering or chemical process); PYP (Physical process); THU (Therapeutic use); BIOL (Biological study); PROC (Process); USES (Uses)
(monounsaturd.; extraction of lipophilic compds. for cosmetic and pharmaceutical uses)
- IT Fatty acids, biological studies
RL: COS (Cosmetic use); PEP (Physical, engineering or chemical process); PYP (Physical process); THU (Therapeutic use); BIOL (Biological study); PROC (Process); USES (Uses)
(polyunsaturd.; extraction of lipophilic compds. for cosmetic and pharmaceutical uses)
- IT Fats and Glyceridic oils, biological studies
RL: COS (Cosmetic use); PEP (Physical, engineering or chemical process); PYP (Physical process); THU (Therapeutic use); BIOL (Biological study); PROC (Process); USES (Uses)
(pumpkin seed; extraction of lipophilic compds. for cosmetic and pharmaceutical uses)
- IT Fatty acids, biological studies
RL: COS (Cosmetic use); PEP (Physical, engineering or chemical process); PYP (Physical process); THU (Therapeutic use); BIOL (Biological study); PROC (Process); USES (Uses)
(saturated; extraction of lipophilic compds. for cosmetic and pharmaceutical uses)
- IT Fats and Glyceridic oils, biological studies
RL: COS (Cosmetic use); PEP (Physical, engineering or chemical process); PYP (Physical process); THU (Therapeutic use); BIOL (Biological study); PROC (Process); USES (Uses)
(saw palmetto; extraction of lipophilic compds. for cosmetic and pharmaceutical uses)
- IT Fatty acids, biological studies
RL: COS (Cosmetic use); PEP (Physical, engineering or chemical process); PYP (Physical process); THU (Therapeutic use); BIOL (Biological study); PROC (Process); USES (Uses)
(unsaturd.; extraction of lipophilic compds. for cosmetic and pharmaceutical uses)
- IT Fats and Glyceridic oils, biological studies
RL: COS (Cosmetic use); PEP (Physical, engineering or chemical process); PYP (Physical process); THU (Therapeutic use); BIOL (Biological study); PROC (Process); USES (Uses)
(vegetable, hydrolyzates; extraction of lipophilic compds. for cosmetic and pharmaceutical uses)
- IT 56-81-5D, Glycerol, ethers 60-33-3, 9,12-Octadecadienoic acid (9Z,12Z)-, biological studies 64-17-5, Ethanol, biological studies 111-02-4, Squalene 112-80-1, Oleic acid, biological studies 115-10-6, Dimethyl ether 463-40-1 472-61-7, Astaxanthin 502-65-8, Lycopene 506-26-3 1406-16-2, Vitamin D 1406-18-4, Vitamin E 6217-54-5 7235-40-7, β -Carotene 10417-94-4 11103-57-4, Vitamin A

RL: COS (Cosmetic use); PEP (Physical, engineering or chemical process);
PYP (Physical process); THU (Therapeutic use); BIOL (Biological study);
PROC (Process); USES (Uses)

(extraction of lipophilic compds. for cosmetic and pharmaceutical uses)

IT 57-13-6, Urea, processes 74-84-0, Ethane, processes 74-85-1, Ethylene,
processes 74-98-6, Propane, processes 106-97-8, Butane, processes
115-07-1, Propylene, processes 811-97-2, 1,1,1,2,-Tetrafluoroethane
2551-62-4, Sulfur hexafluoride 10024-97-2, Nitrous oxide, processes
RL: PEP (Physical, engineering or chemical process); PYP (Physical
process); PROC (Process)

(extraction of lipophilic compds. for cosmetic and pharmaceutical uses)

IT 124-38-9, Carbon dioxide, processes
RL: PEP (Physical, engineering or chemical process); PYP (Physical
process); PROC (Process)

(supercrit.; extraction of lipophilic compds. for cosmetic and
pharmaceutical uses)

RE.CNT 5 THERE ARE 5 CITED REFERENCES AVAILABLE FOR THIS RECORD

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IT 1406-16-2, Vitamin D

RL: COS (Cosmetic use); PEP (Physical, engineering or chemical process);
PYP (Physical process); THU (Therapeutic use); BIOL (Biological study);
PROC (Process); USES (Uses)

(extraction of lipophilic compds. for cosmetic and pharmaceutical uses)

RN 1406-16-2 HCAPLUS

CN Vitamin D (8CI, 9CI) (CA INDEX NAME)

*** STRUCTURE DIAGRAM IS NOT AVAILABLE ***

IT 124-38-9, Carbon dioxide, processes

RL: PEP (Physical, engineering or chemical process); PYP (Physical
process); PROC (Process)

(supercrit.; extraction of lipophilic compds. for cosmetic and
pharmaceutical uses)

RN 124-38-9 HCAPLUS

CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

O=C=O

L79 ANSWER 2 OF 36 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 2003:845155 HCAPLUS

DN 140:82892

ED Entered STN: 29 Oct 2003

TI Calculation of Solid Solubility of Complex Molecules in
Supercritical Carbon Dioxide using a Solution
Model Approach

AU Cheng, Jaw-Shin; Tang, Muoi; Chen, Yan-Ping

CS Department of Chemical Engineering, National Taiwan University, Taipei,
Taiwan

SO Molecular Simulation (2003), 29(12), 749-754

CODEN: MOSIEA; ISSN: 0892-7022

PB Taylor & Francis Ltd.

DT Journal

LA English

CC 68-1 (Phase Equilibria, Chemical Equilibria, and Solutions)

Section cross-reference(s): 65, 69

AB Solid solubility of complex mols. in supercrit. carbon

dioxide was calculated using a solution model approach. These solutes include the biol. compds. of antioxidants, steroids, vitamins, and quinones. The modified AD (m-AD) activity coefficient model proposed by Cheng, J.S. et al. (2002) coupled with the Flory-Huggins equation was employed. The molar volume of the solutes in the **supercrit.** phase and the solid-fluid interaction parameter were optimally fitted for each solid component. Satisfactory results were obtained from the m-AD model with three or four parameters. The overall accuracy in this study is comparably good to that from the conventional equation of state method or the semi-empirical correlation equation. This result suggests a feasible high-pressure solid solubility calcn. with the application of a theor. based solution model.

- ST org compd solid soly **supercrit carbon dioxide**
soln modeling
- IT Activity (thermodynamic)
Antioxidants
Molar volume
Simulation and Modeling, physicochemical
Solid solubility
(calcn. of organic compound solid solubility in **supercrit. carbon dioxide** using solution model approach)
- IT Quinones
Steroids, properties
Vitamins
RL: PEP (Physical, engineering or chemical process); PRP (Properties); PYP (Physical process); PROC (Process)
(calcn. of organic compound solid solubility in **supercrit. carbon dioxide** using solution model approach)
- IT Solvents
(**supercrit.**; calcn. of organic compound solid solubility in **supercrit. carbon dioxide** using solution model approach)
- IT 50-14-6, Vitamin D2 50-29-3, DDT, properties
50-81-7, Ascorbic acid, properties 57-83-0, Progesterone, properties
57-88-5, Cholesterol, properties 58-22-0, Testosterone 67-97-0
, Vitamin D3 71-58-9, Medroxyprogesterone acetate
83-48-7, Stigmasterol 84-65-1, 9,10-Anthraquinone 94-75-7,
2,4-Dichlorophenoxy acetic acid, properties 106-51-4, 1,4-Benzoquinone,
properties 118-74-1, Hexachlorobenzene 121-79-9, Propyl gallate
124-38-9, Carbon dioxide, properties
130-15-4, 1,4-Naphthoquinone 137-66-6, Ascorbyl palmitate 525-82-6,
Flavone 577-85-5, 3-Hydroxyflavone 1166-52-5, Dodecyl gallate
11103-57-4, Vitamin A 22204-53-1, Naproxen
RL: PEP (Physical, engineering or chemical process); PRP (Properties); PYP (Physical process); PROC (Process)
(calcn. of organic compound solid solubility in **supercrit. carbon dioxide** using solution model approach)

RE.CNT 23 THERE ARE 23 CITED REFERENCES AVAILABLE FOR THIS RECORD

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IT 50-14-6, Vitamin D2 67-97-0,

Vitamin D3 124-38-9, Carbon

dioxide, properties

RL: PEP (Physical, engineering or chemical process); PRP (Properties); PYP
 (Physical process); PROC (Process)

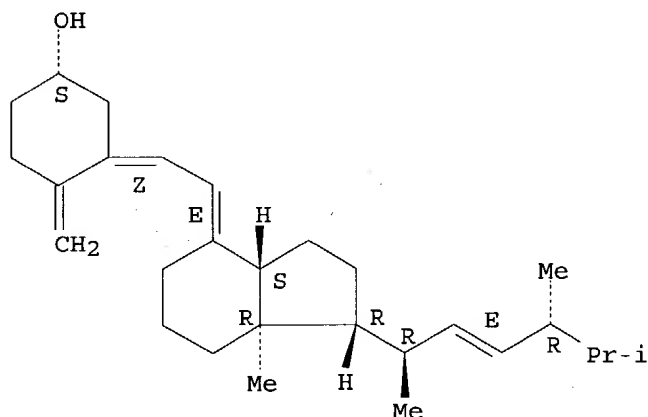
(calcn. of organic compound solid solubility in **supercrit.**

carbon dioxide using solution model approach)

RN 50-14-6 HCAPLUS

CN 9,10-Secoergosta-5,7,10(19),22-tetraen-3-ol, (3 β ,5Z,7E,22E) - (9CI)
 (CA INDEX NAME)

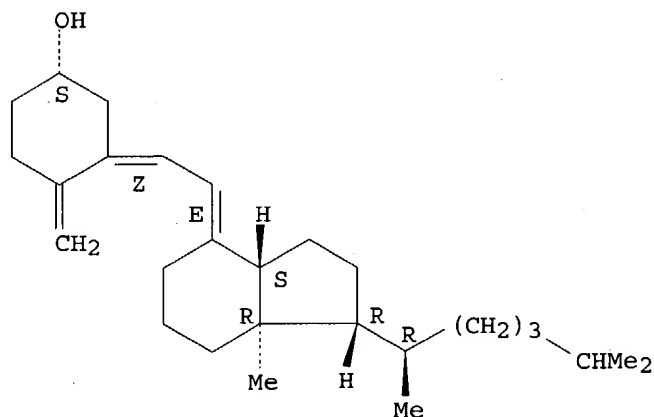
Absolute stereochemistry. Rotation (+).
 Double bond geometry as shown.



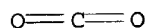
RN 67-97-0 HCAPLUS

CN 9,10-Secocholesta-5,7,10(19)-trien-3-ol, (3 β ,5Z,7E) - (9CI) (CA INDEX NAME)

Absolute stereochemistry.
 Double bond geometry as shown.



RN 124-38-9 HCAPLUS
CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)



L79 ANSWER 3 OF 36 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 2003:651925 HCAPLUS
DN 139:306776
ED Entered STN: 21 Aug 2003
TI Fat-soluble vitamin extraction by analytical **supercritical carbon dioxide**
AU Perretti, Giuseppe; Marconi, Ombretta; Montanari, Luigi; Fantozzi, Paolo
CS Department of Food Science, University of Perugia, Perugia, I-06126, Italy
SO Journal of the American Oil Chemists' Society (2003), 80(7), 629-633
CODEN: JAOCA7; ISSN: 0003-021X
PB AOCS Press
DT Journal
LA English
CC 17-1 (Food and Feed Chemistry)
AB Extraction of fat-soluble vitamins (A, D, E, and β -carotene) by **supercrit. carbon dioxide (SC-CO2)** was tested to replace conventional liquid extraction methods, which require large vols. of organic solvents. **Supercrit. fluid extraction (SFE)** is a rapid extraction technique for fat-soluble vitamins enabling them to be accurately determined using only small vols. of organic solvents. Extns. were performed on ultra-high-temperature sterilized milk, milk powder, pork, liver pate (pate de fois), infant formula, and canned baby food to compare the methods. The proposed method is based on the extraction of fat-soluble vitamins and their esters by using SC-CO2 with methanol as a modifier. HPLC anal. using photometric detection was used for the vitamin anal. The results showed no significant differences between extraction methods. The proposed SFE method appears to be useful as a substitute for the traditional organic solvent method, mainly for vitamin A and γ -tocopherol.
ST food analysis **supercrit** extn fat soluble vitamin
IT Dairy products
Food analysis
Meat
(fat-soluble vitamin extraction by anal. **supercrit. carbon dioxide**)
IT Vitamins
RL: ANT (Analyte); ANST (Analytical study)
(fat-soluble; fat-soluble vitamin extraction by anal. **supercrit. carbon dioxide**)
IT Food
(infant; fat-soluble vitamin extraction by anal. **supercrit. carbon dioxide**)
IT Extraction
(**supercrit.**; fat-soluble vitamin extraction by anal. **supercrit. carbon dioxide**)
IT 59-02-9, α -Tocopherol 67-97-0, Vitamin D3 7235-40-7, β -Carotene 7616-22-0, γ -Tocopherol
RL: ANT (Analyte); ANST (Analytical study)
(fat-soluble vitamin extraction by anal. **supercrit. carbon dioxide**)
IT 124-38-9, Carbon dioxide, analysis
RL: ARU (Analytical role, unclassified); ANST (Analytical study)
(fat-soluble vitamin extraction by anal. **supercrit. carbon**

dioxide)

RE.CNT 23 THERE ARE 23 CITED REFERENCES AVAILABLE FOR THIS RECORD

RE

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IT 67-97-0, Vitamin D3

RL: ANT (Analyte); ANST (Analytical study)

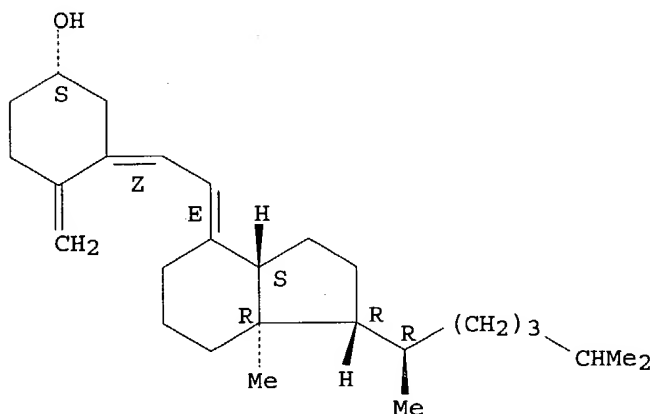
(fat-soluble vitamin extraction by anal. supercrit.

carbon dioxide)

RN 67-97-0 HCAPLUS

CN 9,10-Secocholesta-5,7,10(19)-trien-3-ol, (3 β ,5Z,7E)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.
Double bond geometry as shown.



IT 124-38-9, Carbon dioxide, analysis

RL: ARU (Analytical role, unclassified); ANST (Analytical study)

(fat-soluble vitamin extraction by anal. **supercrit. carbon dioxide**)

RN 124-38-9 HCAPLUS

CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

O=C=O

L79 ANSWER 4 OF 36 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 2003:462432 HCAPLUS

DN 139:58078

ED Entered STN: 17 Jun 2003

TI Preparative **supercritical** fluid **chromatography** as an example of vitamin separation

AU Buss, Vessela

CS Aschaffenburg, Germany

SO Fortschritt-Berichte VDI, Reihe 3: Verfahrenstechnik (2003), 778, i-xiii, 1-138

CODEN: FVVEFK; ISSN: 0178-9503

PB VDI Verlag GmbH

DT Journal

LA German

CC 64-2 (Pharmaceutical Analysis)

AB The use of **supercrit. fluid chromatog.** (SFC) in packed columns was investigated. Zorbax Pro 10-60 CN was applied as stationary phase to sep. in anal. scale several mixts. containing α -tocopherol, the **vitamins D2** and **D3**, or 7-dehydrocholesterol. Pressure and composition of the mobile phase were optimized. A mixture of 20% α -tocopherol and 80% **vitamin D3** was separated by preparative **chromatog.** The separation was carried out with **CO2** and 0.12% (weight) EtOH as modifier at 13.8 MPa and 313.15 K. Purities of >99% **vitamin D3** and 60% α -tocopherol were obtained. Adsorption isothermes of the components were determined by the peak maxima method to predict elution profiles. The construction of a SFC apparatus was described for pressures up to 25 MPa and temps. up to 368.15 K.

ST **supercrit fluid chromatog tocopherol vitamin D**

IT Adsorption

(isotherm; preparative **supercrit. fluid chromatog.** as an example of vitamin separation)

IT Preparative **chromatography**

Simulation and Modeling, physicochemical

Supercritical fluid chromatography

(preparative **supercrit. fluid chromatog.** as an example of vitamin separation)

IT 50-14-6, **Vitamin D2** 67-97-0,

Vitamin D3 434-16-2, 7-Dehydrocholesterol

10191-41-0, D,L- α -Tocopherol

RL: ANT (Analyte); ANST (Analytical study)

(preparative **supercrit. fluid chromatog.** as an example of **vitamin** separation)

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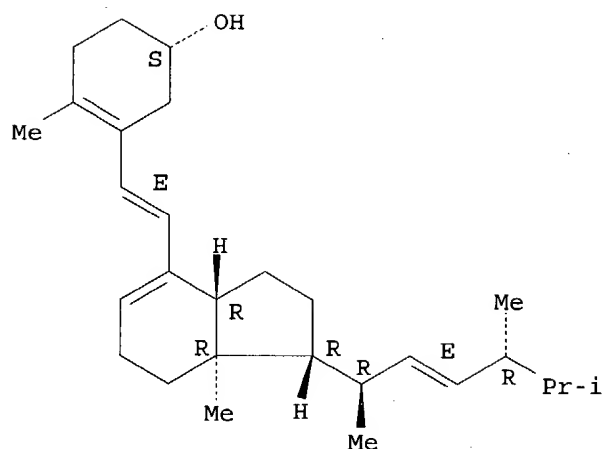
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- (116) Shi, T; Proceedings of the 5th International Symposium on Supercritical Fluids 2000
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- (120) Stanley, B; J Chromatogr A 1997, V761, P41 HCAPLUS
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L79 ANSWER 12 OF 36 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 1998:253034 HCAPLUS
DN 128:312926
ED Entered STN: 04 May 1998
TI Process for the manufacture of a powdery preparation
IN Bausch, Alexander; Peter, Siegfried K. F.; Steiner, Kurt; Stoller, Hans
Jorg; Weidner, Eckhard
PA F. Hoffmann-La Roche A.-G., Switz.
SO PCT Int. Appl., 27 pp.
CODEN: PIXXD2
DT Patent
LA English
IC A61K-914; A61K009-72; A61K310-15
CC 63-6 (Pharmaceuticals)
FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	WO 9816204	A1	19980423	WO 1997-EP5621	19971013 <--
	W: BR, CN, ID, JP				
	RW: AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE				
	CN 1233169	A	19991027	CN 1997-198781	19971013 <--
	EP 952820	A1	19991103	EP 1997-945817	19971013 <--
	R: AT, BE, CH, DE, DK, ES, FR, GB, IT, LI, NL				
	BR 9713258	A	20000328	BR 1997-13258	19971013 <--
	JP 2001505190	T2	20010417	JP 1998-518006	19971013 <--
	EP 1097705	A2	20010509	EP 2000-121676	19971013 <--
	R: AT, BE, CH, DE, DK, ES, FR, GB, IT, LI, NL				
	JP 2001226262	A2	20010821	JP 2000-312886	19971013 <--
	CN 1394597	A	20030205	CN 2000-131456	20001017 <--
	US 2001000143	A1	20010405	US 2000-726876	20001130 <--
	US 2001031282	A1	20011018	US 2000-726875	20001130 <--
	US 2002086059	A1	20020704	US 2001-45270	20011023 <--
PRAI	EP 1996-116420	A	19961014	<--	
	CH 1996-2895	A	19961125	<--	
	US 1997-953952	B1	19971010	<--	
	EP 1997-945817	A3	19971013	<--	
	JP 1998-518006	A3	19971013	<--	
	WO 1997-EP5621	W	19971013	<--	
	US 2000-726876	B1	20001130		

AB The invention concerns a process for the manufacture of powdery active substance selected from the groups of pharmaceuticals, pharmaceutical precursors, diagnostics, fine chems. or carotenoids, especially β -carotene, characterized by (1) dissolving the active substance in di-Me ether under

elevated pressure and temperature conditions, (2) **flash**-decompressing the thus-formed solution in an expansion apparatus, and (3) separating the powdery

solid particles formed in the expansion from the di-Me ether liberated.

Also described is a process for the manufacture of a powdery preparation containing the

active ingredients distributed in a matrix component consisting of at least one adjuvant. Trans- β -carotene 167 g were stirred into 500 g of liquid polyethylene glycol at 90° and the mixture and 350 g di-Me ether were charged into an autoclave. The solution was passed through the inner nozzle and the gas liberated in the spray tower was fed to a cyclone together with the solids. After completion of the spray procedure, the spray tower was opened. Finely divided coppt. of polyethylene glycol and β -carotene were taken off.

ST carotene dimethyl ether adjuvant pulverization

IT Drug delivery systems

(powders; process for manufacture of powdery preparation to improve bioavailability and color intensity)

IT Gums and Mucilages

Hydrocolloids

(process for manufacture of powdery preparation to improve bioavailability

and

color intensity)

IT Carotenes, biological studies

Fats and Glyceridic oils, biological studies

Gelatins, biological studies

Polymers, biological studies

Polyoxyalkylenes, biological studies

Polysaccharides, biological studies

Proteins, general, biological studies

Vitamins

Waxes

RL: THU (Therapeutic use); BIOL (Biological study); USES (Uses)

(process for manufacture of powdery preparation to improve bioavailability

and

color intensity)

IT 79-10-7D, Acrylic acid, esters, polymers 115-10-6, Dimethyl ether

124-38-9, Carbon dioxide, biological studies

439-14-5, Diazepam 723-46-6, Sulfamethoxazole 738-70-5, Trimethoprim

1812-30-2, Bromazepam 4759-48-2, Isotretinoin 7235-40-7,

β -Carotene 7481-89-2, Zalcitabine 9003-39-8, PVP 22204-53-1,

Naproxen 25322-68-3, Polyethylene glycol **32222-06-3**,

Calcitriol 53230-10-7, Mefloquine 59467-70-8, Midazolam 59804-37-4,

Tenoxicam 71320-77-9, Moclobemide 73384-59-5, Ceftriaxone

74103-06-3, Ketorolac 82410-32-0, Ganciclovir 88768-40-5, Cilazapril

96829-58-2, Orlistat 127779-20-8, Saquinavir 128794-94-5,

Mycophenolate mofetil 134308-13-7, Tolcapone 144412-49-7, Lamifiban

147536-97-8, Bosentan 159989-65-8, Nelfinavir mesylate

RL: THU (Therapeutic use); BIOL (Biological study); USES (Uses)

(process for manufacture of powdery preparation to improve bioavailability

and

color intensity)

RE.CNT 7 THERE ARE 7 CITED REFERENCES AVAILABLE FOR THIS RECORD

RE

(1) Basf Ag; DE 2943267 A1 1981 HCAPLUS

(2) Bayer Ag; CH 667203 A5 1988 HCAPLUS

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(6) Peter, S; WO 9615133 A1 1996 HCAPLUS

(7) Weidner, E; WO 9521688 A1 1995 HCAPLUS

IT **124-38-9, Carbon dioxide**, biological studies

32222-06-3, Calcitriol

with a saturator column. With this new mixing device, we could get good resolution for vitamins which are difficult to sep. with pure CO₂.

ST vitamin **supercrit** fluid **chromatog**

IT **Supercritical fluid chromatography**

(solvent mixing device and amperometric microsensor in anal. of vitamins by **supercrit.** fluid **chromatog.**)

IT Vitamins

RL: ANT (Analyte); ANST (Analytical study)

(solvent mixing device and amperometric microsensor in anal. of vitamins by **supercrit.** fluid **chromatog.**)

IT 50-81-7, Ascorbic acid, analysis 59-43-8, Vitamin B1, analysis

59-67-6, Nicotinic acid, analysis 83-88-5, Vitamin B2, analysis

98-92-0, Nicotinamide **1406-16-2, Vitamin D**

1406-18-4, Vitamin E 8059-24-3, Vitamin B6 12001-79-5, Vitamin K

RL: ANT (Analyte); ANST (Analytical study)

(solvent mixing device and amperometric microsensor in anal. of vitamins by **supercrit.** fluid **chromatog.**)

IT **1406-16-2, Vitamin D**

RL: ANT (Analyte); ANST (Analytical study)

(solvent mixing device and amperometric microsensor in anal. of vitamins by **supercrit.** fluid **chromatog.**)

RN 1406-16-2 HCAPLUS

CN Vitamin D (8CI, 9CI) (CA INDEX NAME)

*** STRUCTURE DIAGRAM IS NOT AVAILABLE ***

L79 ANSWER 14 OF 36 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 1997:44341 HCAPLUS

DN 126:51588

ED Entered STN: 21 Jan 1997

TI Solubilities of the Fat-Soluble Vitamins A, D, E, and K in

Supercritical Carbon Dioxide

AU **Johannsen, Monika**; Brunner, Gerd

CS Arbeitsbereich Verfahrenstechnik II, Technical University, Hamburg, D-21073, Germany

SO Journal of Chemical and Engineering Data (1997), 42(1), 106-111

CODEN: JCEAAX; ISSN: 0021-9568

PB American Chemical Society

DT Journal

LA English

CC 68-1 (Phase Equilibriums, Chemical Equilibriums, and Solutions)

Section cross-reference(s): 17, 26

AB Solubilities of eight different species of the fat-soluble vitamins A, D, E, and K in **supercrit. carbon dioxide** were measured at (313, 333, and 353) K and over a pressure range of 20 MPa to 35 MPa. Solubilities have been determined by an anal. method using the direct coupling of an equilibrium cell to a **supercrit.** fluid **chromatog.** system with UV detection. The solubilities of all fat-soluble vitamins in **supercrit. carbon dioxide** under the conditions investigated are in the range of 10 g/kg, except for β -carotene (provitamin A), which is 3 orders of magnitude less soluble. With increasing mol. mass of the vitamin, its solubility

in **supercrit. carbon dioxide** decreases. At

constant temperature, the solubility of all substances increases with increasing d. At

constant d., a rise of temperature results in an increase in solubility. This is caused

by the increasing vapor pressure of the solid.

ST soly fat soluble vitamin **supercrit** solvent; vitamin soly **supercrit carbon dioxide**

IT Vitamins

RL: PRP (Properties)

(fat-soluble; solubilities of fat-soluble vitamins A, D, E, and K in **supercrit. carbon dioxide**)

IT Solubility
(solubilities of fat-soluble vitamins A, D, E, and K in **supercrit. carbon dioxide**)

IT 50-14-6, Vitamin D2 58-27-5, Vitamin K3
67-97-0, Vitamin D3 68-26-8, trans-Retinol
119-13-1, δ -Tocopherol 124-38-9, Carbon
dioxide, properties 7235-40-7, β , β -Carotene
10191-41-0, DL- α -Tocopherol 11104-38-4, Vitamin K1
RL: PRP (Properties)

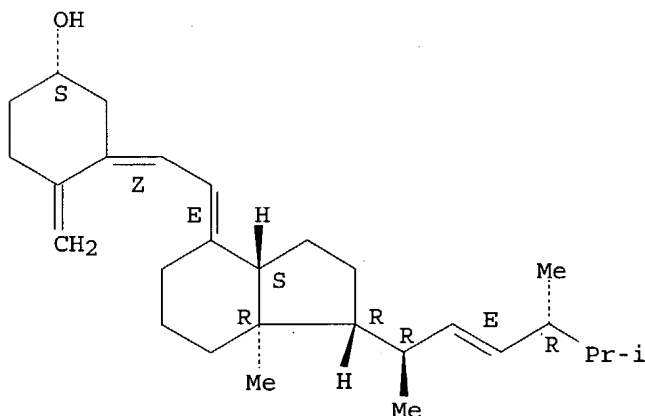
(solubilities of fat-soluble vitamins A, D, E, and K in **supercrit. carbon dioxide**)

IT 50-14-6, Vitamin D2 67-97-0,
Vitamin D3 124-38-9, Carbon
dioxide, properties
RL: PRP (Properties)
(solubilities of fat-soluble vitamins A, D, E, and K in **supercrit. carbon dioxide**)

RN 50-14-6 HCAPLUS

CN 9,10-Secoergosta-5,7,10(19),22-tetraen-3-ol, (3 β ,5Z,7E,22E) - (9CI)
(CA INDEX NAME)

Absolute stereochemistry. Rotation (+).
Double bond geometry as shown.



RN 67-97-0 HCAPLUS

CN 9,10-Secocholesta-5,7,10(19)-trien-3-ol, (3 β ,5Z,7E) - (9CI) (CA INDEX NAME)

Absolute stereochemistry.
Double bond geometry as shown.

chromatog. of fat-soluble vitamins using liquid crystal polysiloxane coated particles)

IT 50-14-6, Vitamin d2 67-97-0,

Vitamin d3 1406-18-4, Vitamin e 11032-49-8, Vitamin

k2 11103-57-4, Vitamin a 11104-38-4, Vitamin k1

RL: ANT (Analyte); ANST (Analytical study)

(packed capillary column **supercrit.** fluid **chromatog.**

. of fat-soluble **vitamins** using liquid crystal polysiloxane coated particles)

IT 50-14-6, Vitamin d2 67-97-0,

Vitamin d3

RL: ANT (Analyte); ANST (Analytical study)

(packed capillary column **supercrit.** fluid **chromatog.**

. of fat-soluble **vitamins** using liquid crystal polysiloxane coated particles)

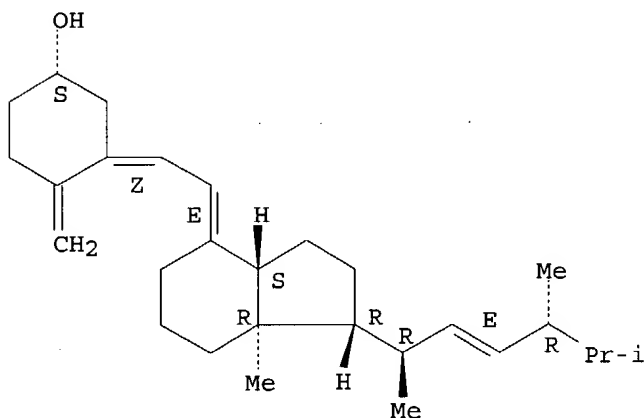
RN 50-14-6 HCAPLUS

CN 9,10-Secoergosta-5,7,10(19),22-tetraen-3-ol, (3 β ,5Z,7E,22E) - (9CI)

(CA INDEX NAME)

Absolute stereochemistry. Rotation (+).

Double bond geometry as shown.



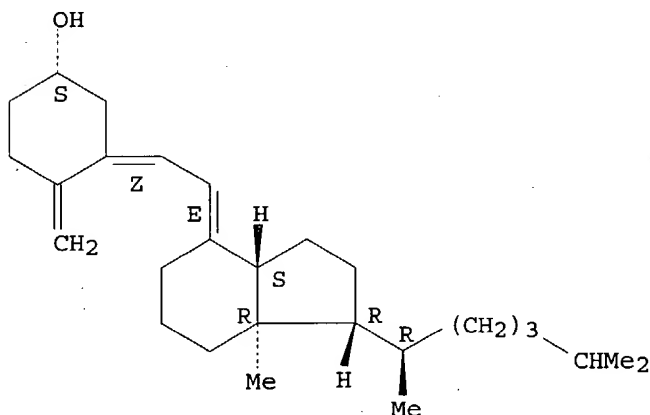
RN 67-97-0 HCAPLUS

CN 9,10-Secocholesta-5,7,10(19)-trien-3-ol, (3 β ,5Z,7E) - (9CI) (CA INDEX

NAME)

Absolute stereochemistry.

Double bond geometry as shown.



L79 ANSWER 16 OF 36 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 1995:956938 HCAPLUS
DN 124:105153
ED Entered STN: 01 Dec 1995
TI Applications of reversed-phase high performance liquid
chromatography using enhanced-fluidity liquid mobile phases
AU Lee, Stephen T.; Olesik, Susan V.; Fields, Steven M.
CS Department of Chemistry, The Ohio State University, Columbus, OH, 43210,
USA
SO Journal of Microcolumn Separations (1995), 7(5), 477-83
CODEN: JMSEJ; ISSN: 1040-7685
PB Wiley
DT Journal
LA English
CC 80-4 (Organic Analytical Chemistry)
Section cross-reference(s): 51, 64
AB Enhanced-fluidity liquid mobile phases (methanol/H2O/CO2) were
used as eluents in reversed-phase HPLC. The low pressure drop across the
column allowed serial connection of micro-scale columns to achieve the
efficient separation of a coal tar sample. Other applications such as the
separation of fat soluble vitamins and probucol and related compds. are shown.
ST vitamin sepn reversed phase HPLC; reversed phase HPLC enhanced fluidity;
coal tar reversed phase HPLC; drug analysis reversed phase HPLC; liq
chromatog enhanced fluidity mobile phase
IT Pharmaceutical analysis
(coal tar and vitamins and drugs separation by reversed-phase high
performance liquid **chromatog.** using enhanced-fluidity liquid
mobile phases)
IT Tar
RL: ANT (Analyte); ANST (Analytical study)
(coal, coal tar and vitamins and drugs separation by reversed-phase high
performance liquid **chromatog.** using enhanced-fluidity liquid
mobile phases)
IT Vitamins
RL: ANT (Analyte); ANST (Analytical study)
(fat-soluble, coal tar and vitamins and drugs separation by reversed-phase
high
performance liquid **chromatog.** using enhanced-fluidity liquid
mobile phases)
IT **Chromatography, column and liquid**
(**high-performance reversed-phase**
, coal tar and vitamins and drugs separation by **reversed-**
phase high performance liquid
chromatog. using enhanced-fluidity liquid mobile phases)
IT Aromatic hydrocarbons, analysis
RL: ANT (Analyte); ANST (Analytical study)
(polycyclic, coal tar and vitamins and drugs separation by reversed-phase
high performance liquid **chromatog.** using enhanced-fluidity liquid
mobile phases)
IT 50-14-6, Ergocalciferol 50-32-8, (Benzo[a]pyrene), analysis
53-70-3, Dibenz[a,h]anthracene 56-55-3, Benzo[a]anthracene
67-97-0, Cholecalciferol 68-26-8, trans-Retinol
71-43-2, Benzene, analysis 85-01-8, Phenanthrene, analysis 86-73-7,
Fluorene 91-20-3, Naphthalene, analysis 116-31-4, trans-Retinal
120-12-7, Anthracene, analysis 127-47-9, Retinol acetate 128-37-0,
BHT, analysis 128-38-1 129-00-0, Pyrene, analysis 191-24-2,
Benzo[ghi]perylene 193-39-5, (Indeno[1,2,3-cd]pyrene) 205-99-2,
Benzo[b]fluoranthene 206-44-0, Fluoranthene 207-08-9,
(Benzo[k]fluoranthene) 208-96-8, Acenaphthylene 218-01-9, Chrysene
2455-14-3 10191-41-0 11104-38-4, Vitamin K1 23288-49-5, Probuco
26067-78-7 51571-18-7 52225-20-4 129895-82-5
RL: ANT (Analyte); ANST (Analytical study)

(coal tar and vitamins and drugs separation by reversed-phase high performance liquid **chromatog.** using enhanced-fluidity liquid mobile phases)

IT 50-14-6, Ergocalciferol 67-97-0, Cholecalciferol

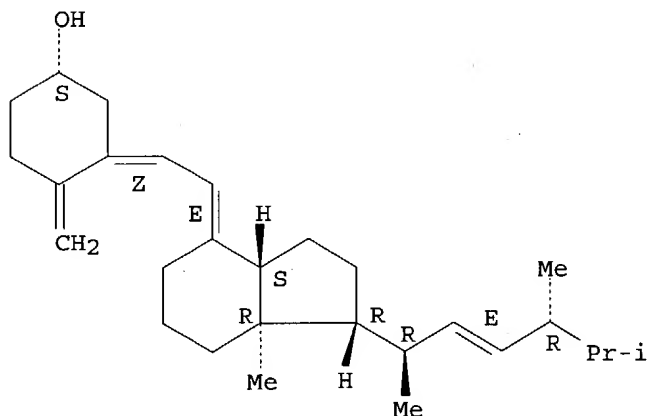
RL: ANT (Analyte); ANST (Analytical study)

(coal tar and vitamins and drugs separation by reversed-phase high performance liquid **chromatog.** using enhanced-fluidity liquid mobile phases)

RN 50-14-6 HCAPLUS

CN 9,10-Secoergosta-5,7,10(19),22-tetraen-3-ol, (3 β ,5Z,7E,22E)- (9CI)
(CA INDEX NAME)

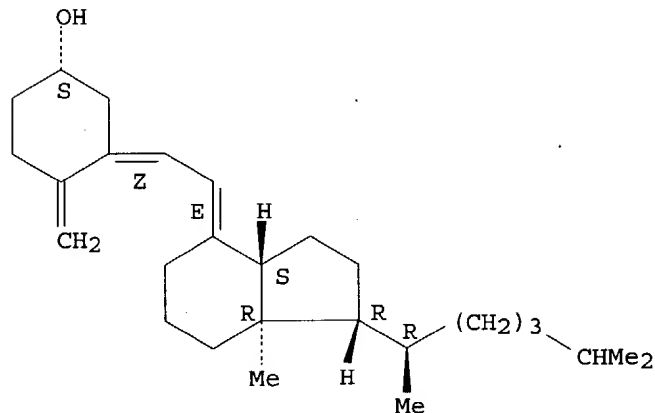
Absolute stereochemistry. Rotation (+).
Double bond geometry as shown.



RN 67-97-0 HCAPLUS

CN 9,10-Secocholesta-5,7,10(19)-trien-3-ol, (3 β ,5Z,7E)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.
Double bond geometry as shown.



L79 ANSWER 17 OF 36 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 1995:319449 HCAPLUS

DN 122:104187

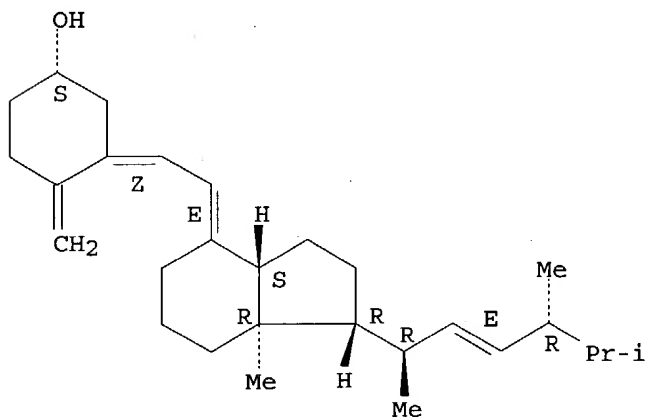
ED Entered STN: 28 Jan 1995

TI Quantitative analysis of marine oils by capillary **supercritical**
fluid **chromatography**

- AU Staby, A.; Borch-Jensen, C.; Balchen, S.; Mollerup, J.
 CS Dep. Chem. Eng., Technical Univ. Denmark, Lyngby, 2800, Den.
 SO Chromatographia (1994), 39(11/12), 697-705
 CODEN: CHRGB7; ISSN: 0009-5893
 PB Vieweg
 DT Journal
 LA English
 CC 17-1 (Food and Feed Chemistry)
 AB **Supercrit. fluid chromatog.** anal. methods have been
 employed in the examination of several marine oils for the group separation of
 free fatty acids, retinol, ergocalciferol, **cholecalciferol**, squalene,
 tocopherols, cholesterol, wax esters, diacylglycerols, cholesteryl esters,
 and triacylglycerols. The oils were derived from characteristic species
 including shark, seal, edible and trash fish. The **supercrit.**
fluid chromatog. (SFC) method used for the separation of the liqs.
 utilize **carbon dioxide** as the mobile phase, a
 non-polar capillary column, and flame ionization detection. The SFC
 methods have proved capable of making a considerable contribution to the
 continuing investigations into the structure and composition of marine oils.
 Furthermore SFC analyses, with their very simple sample preparation
 requirements, may serve as alternatives or supplements to the existing
 range of **chromatog.** and non-**chromatog.** anal. methods
 used in the examination of these oils.
 ST fish oil capillary **supercrit fluid chromatog**; marine
 oil capillary **supercrit fluid chromatog**
 IT Fats and Glyceridic oils
 RL: AMX (Analytical matrix); ANST (Analytical study)
 (basking shark; determination of marine oil constituents by capillary
supercrit. fluid chromatog.)
 IT Cod-liver oil
 RL: AMX (Analytical matrix); ANST (Analytical study)
 (determination of marine oil constituents by capillary **supercrit.**
fluid chromatog.)
 IT Fatty acids, analysis
 RL: ANT (Analyte); ANST (Analytical study)
 (determination of marine oil constituents by capillary **supercrit.**
fluid chromatog.)
 IT Glycerides, analysis
 RL: ANT (Analyte); ANST (Analytical study)
 (determination of marine oil constituents by capillary **supercrit.**
fluid chromatog.)
 IT Tocopherols
 RL: ANT (Analyte); ANST (Analytical study)
 (determination of marine oil constituents by capillary **supercrit.**
fluid chromatog.)
 IT Waxes and Waxy substances
 RL: ANT (Analyte); ANST (Analytical study)
 (determination of marine oil constituents by capillary **supercrit.**
fluid chromatog.)
 IT **Chromatography, supercritical fluid**
 (capillary, determination of marine oil constituents by
capillary supercrit. fluid chromatog.)
 IT Glycerides, analysis
 RL: ANT (Analyte); ANST (Analytical study)
 (di-, determination of marine oil constituents by capillary **supercrit**
. fluid chromatog.)
 IT Vitamins
 RL: ANT (Analyte); ANST (Analytical study)
 (fat-soluble, determination of marine oil constituents by capillary
supercrit. fluid chromatog.)
 IT Fats and Glyceridic oils
 RL: AMX (Analytical matrix); ANST (Analytical study)

- (fish, determination of marine oil constituents by capillary **supercrit**
 . fluid **chromatog.**)
- IT Fats and Glyceridic oils
 RL: AMX (Analytical matrix); ANST (Analytical study)
 (herring, determination of marine oil constituents by capillary
supercrit. fluid chromatog.)
- IT Fats and Glyceridic oils
 RL: AMX (Analytical matrix); ANST (Analytical study)
 (mackerel, determination of marine oil constituents by capillary
supercrit. fluid chromatog.)
- IT Fats and Glyceridic oils
 RL: AMX (Analytical matrix); ANST (Analytical study)
 (sand eel, determination of marine oil constituents by capillary
supercrit. fluid chromatog.)
- IT Fats and Glyceridic oils
 RL: AMX (Analytical matrix); ANST (Analytical study)
 (seal, determination of marine oil constituents by capillary **supercrit**
 . fluid **chromatog.**)
- IT Fats and Glyceridic oils
 RL: AMX (Analytical matrix); ANST (Analytical study)
 (tuna, determination of marine oil constituents by capillary **supercrit**
 . fluid **chromatog.**)
- IT 50-14-6, Ergocalciferol 57-88-5, Cholesterol, analysis
 57-88-5D, Cholesterol, esters 67-97-0, Cholecalciferol
 68-26-8, Retinol 111-02-4, Squalene 119-13-1, 8-Tocopherol
 RL: ANT (Analyte); ANST (Analytical study)
 (determination of marine oil constituents by capillary **supercrit.**
 fluid **chromatog.**)
- IT 50-14-6, Ergocalciferol 67-97-0, Cholecalciferol
 RL: ANT (Analyte); ANST (Analytical study)
 (determination of marine oil constituents by capillary **supercrit.**
 fluid **chromatog.**)
- RN 50-14-6 HCAPLUS
 CN 9,10-Secoergosta-5,7,10(19),22-tetraen-3-ol, (3 β ,5Z,7E,22E) - (9CI)
 (CA INDEX NAME)

Absolute stereochemistry. Rotation (+).
 Double bond geometry as shown.

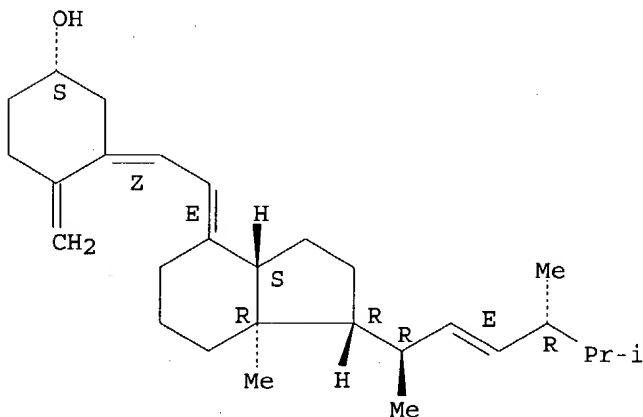


- RN 67-97-0 HCAPLUS
 CN 9,10-Secocholesta-5,7,10(19)-trien-3-ol, (3 β ,5Z,7E) - (9CI) (CA INDEX
 NAME)

Absolute stereochemistry.
 Double bond geometry as shown.

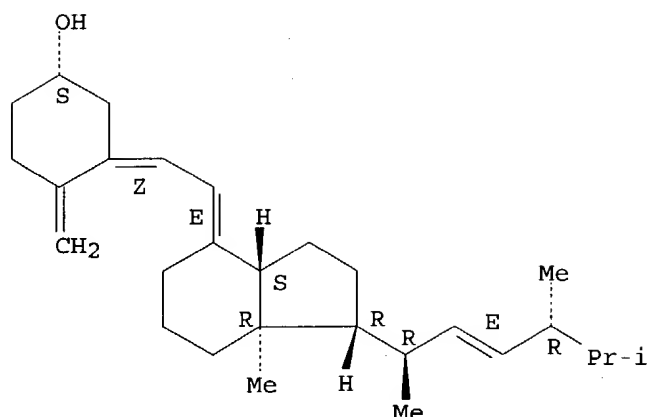
- groups plays an important role.
- ST **supercrit fluid chromatog** retention behavior;
stationary phase **supercrit fluid chromatog**; xanthine
supercrit fluid chromatog; vitamin **supercrit**
fluid chromatog
- IT **Silica gel, compounds**
RL: ANST (Analytical study)
(alkylated, as stationary phase in **supercrit. fluid**
chromatog., retention behavior of)
- IT **Silica gel, compounds**
RL: ANST (Analytical study)
(aminopropylated, as stationary phase in **supercrit.**
fluid chromatog., retention behavior of)
- IT **Silica gel, compounds**
RL: ANST (Analytical study)
(cyanopropylated, as stationary phase in **supercrit. fluid**
chromatog., retention behavior of)
- IT Vitamins
RL: ANT (Analyte); ANST (Analytical study)
(fat-soluble, **chromatog.** of, **supercrit.-fluid**,
stationary phases in)
- IT **Chromatography, gas**
(**supercrit.**, of fat-soluble vitamins and xanthines)
- IT **Chromatography, gas**
(**supercrit.**, stationary phases, silica
gel derivs. as, retention behavior of)
- IT 50-14-6, Vitamin D2 58-08-2, Caffeine,
analysis 58-55-9, Theophylline, analysis 68-26-8, Vitamin A
69-89-6D, derivs. 83-67-0, Theobromine 1406-18-4, Vitamin E
RL: ANT (Analyte); ANST (Analytical study)
(**chromatog.** of, **supercrit.-fluid**, stationary phases
in)
- IT 50-14-6, Vitamin D2
RL: ANT (Analyte); ANST (Analytical study)
(**chromatog.** of, **supercrit.-fluid**, stationary phases
in)
- RN 50-14-6 HCAPLUS
- CN 9,10-Secoergosta-5,7,10(19),22-tetraen-3-ol, (3 β ,5Z,7E,22E)- (9CI)
(CA INDEX NAME)

Absolute stereochemistry. Rotation (+).
Double bond geometry as shown.



DN 99:186740
 ED Entered STN: 12 May 1984
 TI The use of modifiers in **supercritical fluid chromatography with carbon dioxide**
 AU Board, R.; McManigill, D.; Weaver, H.; Gere, D.
 CS Hewlett-Packard Co., Avondale, PA, 19311, USA
 SO ChemSA (1983), (June), 12, 21-2, 24
 CODEN: CHEMDU; ISSN: 0379-4687
 DT Journal
 LA English
 CC 80-4 (Organic Analytical Chemistry)
 Section cross-reference(s): 2, 64
 AB The effects of modifiers, such as MeOH, iso-PrOH, and THF, in **supercrit. fluid chromatog** with CO₂ as the mobile phase were studied. The retention times of di-Me terephthalate, di-Me isophthalate, and di-Me phthalate on a RP-18 bonded phase column were altered >50 times by changing the modifier concentration from 0-1% in the CO₂ mobile phase. The elution of ubiquinones and fat soluble vitamins from a reversed phase RP-18 column was possible only after the addition of MeOH to the mobile phase. With a MeOH modifier concentration of 1.5%, progesterone was separated from estrone on a reversed phase RP-8 column at 34°. At MeOH concns. >1%, the retention times of nicotine and caffeine on a polystyrene reversed phase column were affected significantly. Stationary phase-modifier interaction was the predominant mechanism for changes in retention times at modifier concns. ≤1%.
 ST **supercrit gas chromatog** modifier effect; methanol modifier **supercrit gas chromatog**; isopropanol modifier **supercrit gas chromatog**; THF modifier **supercrit gas chromatog**; terephthalate dimethyl **supercrit gas chromatog**; isophthalate dimethyl **supercrit gas chromatog**; phthalate dimethyl **supercrit gas chromatog**; ubiquinone **supercrit gas chromatog**; vitamin **supercrit gas chromatog**; progesterone **supercrit gas chromatog**; estrone **supercrit gas chromatog**
 IT **Chromatography, gas**
 (supercrit., with carbon dioxide mobile phase, modifier effects in)
 IT 67-56-1, uses and miscellaneous 67-63-0, uses and miscellaneous
 109-99-9, uses and miscellaneous
 RL: ANST (Analytical study); USES (Uses)
 (as modifier in **supercrit. fluid chromatog.** with carbon dioxide mobile phase)
 IT 50-14-6 54-11-5 57-83-0, analysis 58-08-2, analysis
 68-26-8 120-61-6 131-11-3 303-95-7 303-97-9 606-06-4 1065-31-2
 1406-18-4 1459-93-4 2394-68-5
 RL: ANST (Analytical study)
 (separation of, by **supercrit. fluid chromatog.** with carbon dioxide mobile phase, modifier effects on)
 IT 50-14-6
 RL: ANST (Analytical study)
 (separation of, by **supercrit. fluid chromatog.** with carbon dioxide mobile phase, modifier effects on)
 RN 50-14-6 HCAPLUS
 CN 9,10-Secoergosta-5,7,10(19),22-tetraen-3-ol, (3β,5Z,7E,22E)- (9CI)
 (CA INDEX NAME)

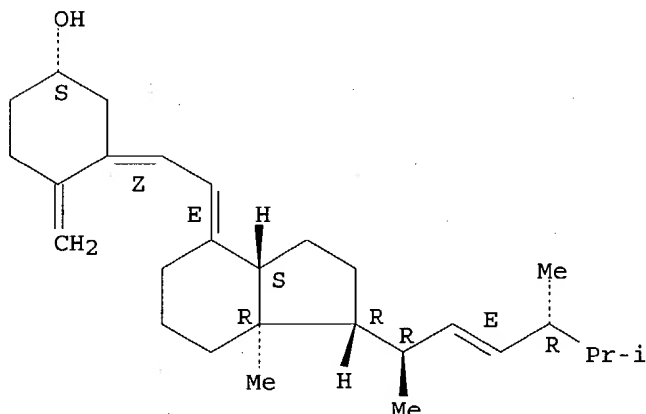
Absolute stereochemistry. Rotation (+).
 Double bond geometry as shown.



L79 ANSWER 21 OF 36 HCAPLUS COPYRIGHT 2004 ACS on STN
 AN 1981:495426 HCAPLUS
 DN 95:95426
 ED Entered STN: 12 May 1984
 TI Possibility of using the **carbon dioxide** extract of yeast residues of grape wines for cosmetics
 AU Kupriyanova, L. A.; Shcherbakov, V. G.; Kalistratova, T. P.; Pekhov, A. V.; Cherevatyi, V. S.; Lebedeva, A. I.
 CS Krasnodar. Nauchno-Issled. Inst. Pishchevoi Prom., Krasnodar, USSR
 SO Izvestiya Vysshikh Uchebnykh Zavedenii, Pishchevaya Tekhnologiya (1981), (3), 40-2
 CODEN: IVUPA8; ISSN: 0579-3009
 DT Journal
 LA Russian
 CC 16-3 (Fermentations)
 Section cross-reference(s): 62
 AB The CO₂ extract of wine lees contains 1% **provitamin D** (calciferol), 190 mg% provitamin A (carotenoid), 30 mg% vitamin E (tocopherol), and 35% essential fatty acids. Palmitate and linoleate constitute 32.42 and 34.38%, resp., of the total fatty acid content. The CO₂ extract maintains its biol. activity at 20° in the dark for 6 mo and may be used as a supplement in the cosmetic industry.
 ST wine yeast ext cosmetics; **carbon dioxide** yeast ext cosmetics
 IT Cosmetics
 (carbon dioxide extract of wine yeast in)
 IT Fatty acids, biological studies
 Vitamins
 RL: BIOL (Biological study)
 (of yeast extract, cosmetics in relation to)
 IT Yeast
 (wine, **carbon dioxide** extract of, as cosmetics ingredient)
 IT 124-38-9, biological studies
 RL: BIOL (Biological study)
 (yeast extract of, as cosmetics ingredient)
 IT 124-38-9, biological studies
 RL: BIOL (Biological study)
 (yeast extract of, as cosmetics ingredient)
 RN 124-38-9 HCAPLUS
 CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

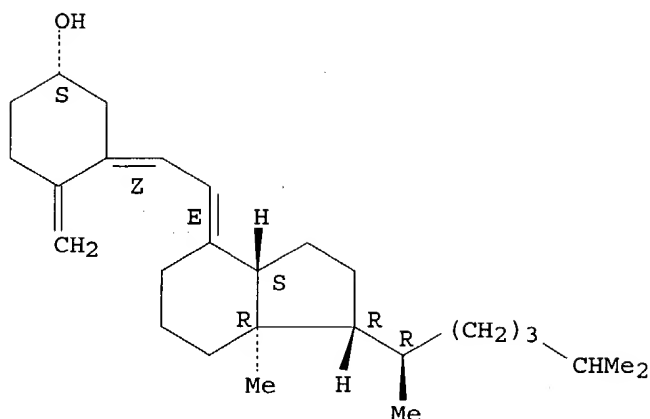
- CN 9,10-Secoergosta-5,7,10(19),22-tetraen-3-ol, (3 β ,5Z,7E,22E)- (9CI)
(CA INDEX NAME)

Absolute stereochemistry. Rotation (+).
Double bond geometry as shown.



CN 9,10-Secocholesta-5,7,10(19)-trien-3-ol, (3 β ,5Z,7E)- (9CI) (CA INDEX
NAME)

Absolute stereochemistry.
Double bond geometry as shown.



L79 ANSWER 5 OF 36 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 2002:593413 HCAPLUS
DN 137:278027
ED Entered STN: 09 Aug 2002
TI Development of methods for the determination of vitamins A, E and
β-carotene in processed foods based on **supercritical** fluid
extraction: a collaborative study
AU Mathiasson, L.; Turner, C.; Berg, H.; Dahlberg, L.; Theobald, A.; Anklaam,
E.; Ginn, R.; Sharman, M.; Ulberth, F.; Gabernig, R.
CS Department of Analytical Chemistry, Lund University, Lund, S-221 00, Swed.
SO Food Additives and Contaminants (2002), 19(7), 632-646
CODEN: FACOEB; ISSN: 0265-203X
PB Taylor & Francis Ltd.
DT Journal
LA English
CC 17-1 (Food and Feed Chemistry)
AB New methodologies based on **supercrit.** fluid extraction (SFE) have
been developed for the determination of fat-soluble vitamins in processed
foods. The
results obtained so far indicate that SFE is well suited to extraction of
fat-soluble vitamins from food products, although validation work is required
to establish accuracy and precision. The vitamins investigated were A, E
and β-carotene, and the processed foods were UHT milk, milk powder,
minced meat, liver paste, infant formula, canned baby food and margarine.
Extraction equipment employed analyte collection on either a solid-phase trap
or in a solvent. After extraction, the samples were saponified and the
vitamins
determined using reversed-phase liquid **chromatog.** with **UV** or
fluorescence detection. Sample throughput was at least 12 samples day⁻¹,
i.e. at least twice the number achievable with a conventional extraction
methodol.
The detection limits for the vitamins in different processed foods were
well below 0.1 μg g⁻¹. Recoveries (in comparison with vitamin levels
obtained using conventional solvent extraction) were close to 100% for
experienced personal with access to modern automatic equipment. To reach
this level, it was necessary to protect the vitamins with an antioxidant
during the different steps of the anal. procedure, to add methanol or
ethanol to the extraction cell to facilitate the analyte extraction from the
food
matrix, and when using a solid-phase trap, to employ a fractionated
extraction-elution procedure to prevent breakthrough losses. The developed
methods were tested in a validation exercise between five labs., which had
taken part in the method development, and in an intercomparison between 10
labs. including labs. with less experience of vitamin determination. The

within-laboratory RSD was generally $\leq 11\%$. The average of the between-laboratory relative standard deviation (RSD) was about 23% in the validation, and increased to about 40% in the intercomparison. Ruggedness tests performed at different steps of the project showed that different types and models of equipment did not give large differences in recoveries. Thus, the increasing RSD can largely be ascribed to differences in experience in vitamin anal. of the participants.

ST vitamin detn food **supercrit** fluid extn; carotene detn food **supercrit** fluid extn

IT Food analysis
Margarine
Milk analysis
Reversed phase HPLC
Sample preparation
Saponification
(determination of vitamins A, E and β -carotene in processed foods based on **supercrit.** fluid extraction)

IT Vitamins
RL: ANT (Analyte); ANST (Analytical study)
(fat-soluble; determination of vitamins A, E and β -carotene in processed foods based on **supercrit.** fluid extraction)

IT Milk substitutes
(human; determination of vitamins A, E and β -carotene in processed foods based on **supercrit.** fluid extraction)

IT Canned foods
(infant; determination of vitamins A, E and β -carotene in processed foods based on **supercrit.** fluid extraction)

IT Meat
(liver, paste; determination of vitamins A, E and β -carotene in processed foods based on **supercrit.** fluid extraction)

IT Meat
(pork; determination of vitamins A, E and β -carotene in processed foods based on **supercrit.** fluid extraction)

IT Extraction
(**supercrit.**; determination of vitamins A, E and β -carotene in processed foods based on **supercrit.** fluid extraction)

IT 50-81-7, Ascorbic acid, uses
RL: ARG (Analytical reagent use); ANST (Analytical study); USES (Uses)
(antioxidant; determination of vitamins A, E and β -carotene in processed foods based on **supercrit.** fluid extraction)

IT 50-14-6, Vitamin D2 59-02-9,
 α -Tocopherol 67-97-0, Vitamin D3
68-26-8, all-trans-Retinol 119-13-1, δ -Tocopherol 148-03-8,
 β -Tocopherol 1406-18-4, Vitamin E 7235-40-7, β -Carotene
7616-22-0, γ -Tocopherol 11103-57-4, Vitamin A
RL: ANT (Analyte); ANST (Analytical study)
(determination of vitamins A, E and β -carotene in processed foods based on **supercrit.** fluid extraction)

IT 124-38-9, Carbon dioxide, analysis
RL: ARU (Analytical role, unclassified); ANST (Analytical study)
(determination of vitamins A, E and β -carotene in processed foods based on **supercrit.** fluid extraction)

IT 64-17-5, Ethanol, uses 67-56-1, Methanol, uses
RL: ARG (Analytical reagent use); ANST (Analytical study); USES (Uses)
(modifier; determination of vitamins A, E and β -carotene in processed foods based on **supercrit.** fluid extraction)

RE.CNT 28 THERE ARE 28 CITED REFERENCES AVAILABLE FOR THIS RECORD
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- (27) Zamarreno, M; Journal of Chromatography 1992, V623, P69
- (28) Zonta, F; Journal of Chromatography 1982, V246, P105 HCAPLUS

IT 50-14-6, Vitamin D2 67-97-0,

Vitamin D3

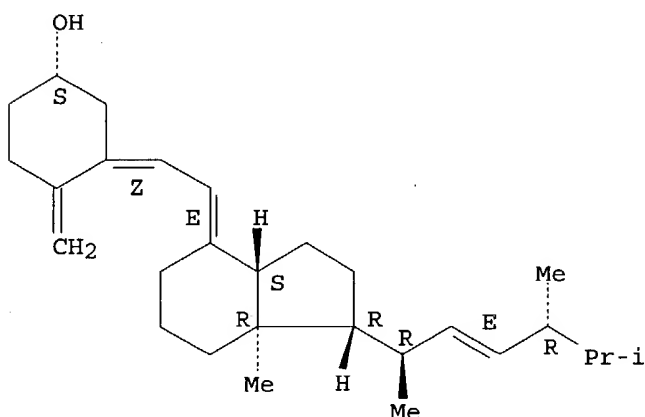
RL: ANT (Analyte); ANST (Analytical study)

(determination of vitamins A, E and β -carotene in processed foods based on supercrit. fluid extraction)

RN 50-14-6 HCAPLUS

CN 9,10-Secoergosta-5,7,10(19),22-tetraen-3-ol, (3 β ,5Z,7E,22E) - (9CI)
(CA INDEX NAME)

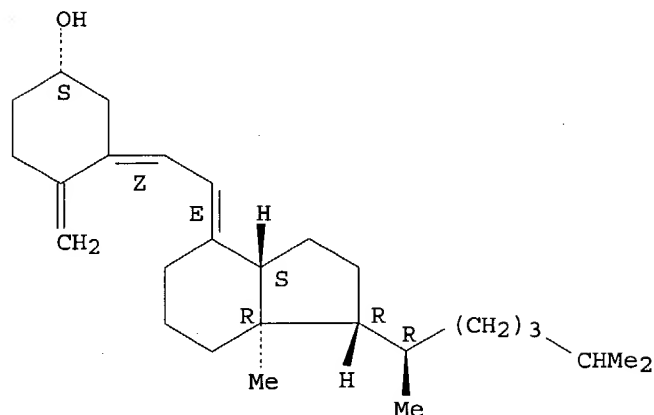
Absolute stereochemistry. Rotation (+).
Double bond geometry as shown.



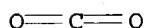
RN 67-97-0 HCAPLUS

CN 9,10-Secocholesta-5,7,10(19)-trien-3-ol, (3 β ,5Z,7E) - (9CI) (CA INDEX

Absolute stereochemistry.
Double bond geometry as shown.



IT 124-38-9, Carbon dioxide, analysis
RL: ARU (Analytical role, unclassified); ANST (Analytical study)
(determination of vitamins A, E and β -carotene in processed foods based on
supercrit. fluid extraction)
RN 124-38-9 HCAPLUS
CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)



L79 ANSWER 6 OF 36 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 2001:780606 HCAPLUS
DN 135:303109
ED Entered STN: 26 Oct 2001
TI Method for producing foodstuffs and/or feedstuffs by removing undesired
lipid constituents using **supercritical CO2** and by
introducing desired lipophilic substances using compressed **CO2**
IN Heidlas, Juergen; Stork, Kurt; Zhang, Zhengfeng; Ober, Martin;
Wiesmueller, Johann; Obersteiner, Johann
PA Degussa A.-G., Germany
SO PCT Int. Appl., 23 pp.
CODEN: PIXXD2
DT Patent
LA German
IC ICM A23L001-015
ICS A23K001-16; A23G001-02; A23G001-00; A23L001-10; A23L001-164
CC 17-4 (Food and Feed Chemistry)
FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
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PI	WO 2001078525	A1	20011025	WO 2001-EP3984	20010406
	W:	AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM			
	RW:	GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW, AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR, BF			

BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG
 DE 10018606 A1 20011025 DE 2000-10018606 20000414
 EP 1272051 A1 20030108 EP 2001-940300 20010406

R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT,
 IE, SI, LT, LV, FI, RO, MK, CY, AL, TR

PRAI DE 2000-10018606 A 20000414
 WO 2001-EP3984 W 20010406

AB The invention relates to a method for producing foods and/or feeds having improved physiol. properties by using dry compressed gases. According to the invention, lipid constituents, in particular triglycerides and other fat accompanying substances, are removed from the food matrix by means of extraction involving the use of **supercrit. CO2** optionally having a proportion of up to 50 weight% propane. The extracted lipid constituents are then separated using known methods, and the extracted food matrix

lipophilic constituents, particularly nutrition-physiol. enhancing additives, are homogeneously introduced using compressed **CO2** optionally having a proportion of up to 50 weight% propane. The particular implementation of the method at preferred pressures ranging from 100 to 800 bar and at temps. ranging from 31 to 100°C enables the specific introduction of, in particular, fat-soluble vitamins, their derivs. or precursors as well as polyunsatd. fatty acids, lipoic acids or sterol derivs. In this manner, foods or feeds can be obtained such as cereals with polyunsatd. fatty acids, cocoa and egg-yolk products which, in the form of functional foods, take the modified nutritional properties into account.

ST food feed processing lipid **supercrit carbon dioxide**

IT Vitamins

RL: FFD (Food or feed use); BIOL (Biological study); USES (Uses)
 (fat-soluble; method for producing foodstuffs and/or feedstuffs by removing undesired lipid constituents using **supercrit. CO2** and by introducing desired lipophilic substances using compressed **CO2**)

IT Egg yolk

(fats; method for producing foodstuffs and/or feedstuffs by removing undesired lipid constituents using **supercrit. CO2** and by introducing desired lipophilic substances using compressed **CO2**)

IT Cereal (grain)

Food functional properties

Lipophilicity

Oatmeal

(method for producing foodstuffs and/or feedstuffs by removing undesired lipid constituents using **supercrit. CO2** and by introducing desired lipophilic substances using compressed **CO2**)

IT Cocoa butter

Sterols

RL: FFD (Food or feed use); BIOL (Biological study); USES (Uses)
 (method for producing foodstuffs and/or feedstuffs by removing undesired lipid constituents using **supercrit. CO2** and by introducing desired lipophilic substances using compressed **CO2**)

IT Fats and Glyceridic oils, processes

RL: REM (Removal or disposal); PROC (Process)

(method for producing foodstuffs and/or feedstuffs by removing undesired lipid constituents using **supercrit. CO2** and by introducing desired lipophilic substances using compressed **CO2**)

IT Glycerides, processes

RL: REM (Removal or disposal); PROC (Process)

(method for producing foodstuffs and/or feedstuffs by removing

process); BIOL (Biological study); PROC (Process); USES (Uses)
(method for producing foodstuffs and/or feedstuffs by removing
undesired lipid constituents using **supercrit. CO2**
and by introducing desired lipophilic substances using compressed
CO2)

IT 124-38-9, Carbon dioxide, biological studies

RL: FFD (Food or feed use); PEP (Physical, engineering or chemical
process); BIOL (Biological study); PROC (Process); USES (Uses)
(**supercrit.**; method for producing foodstuffs and/or
feedstuffs by removing undesired lipid constituents using
supercrit. CO2 and by introducing desired lipophilic
substances using compressed CO2)

RE.CNT 10 THERE ARE 10 CITED REFERENCES AVAILABLE FOR THIS RECORD
RE

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Foods - 5th Ed" 1992, P40
- (10) Vecsei, R; DE 3540544 A 1987

IT 1406-16-2, Vitamin D

RL: FFD (Food or feed use); BIOL (Biological study); USES (Uses)
(method for producing foodstuffs and/or feedstuffs by removing
undesired lipid constituents using **supercrit. CO2**
and by introducing desired lipophilic substances using compressed
CO2)

RN 1406-16-2 HCAPLUS

CN Vitamin D (8CI, 9CI) (CA INDEX NAME)

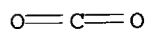
*** STRUCTURE DIAGRAM IS NOT AVAILABLE ***

IT 124-38-9, Carbon dioxide, biological studies

RL: FFD (Food or feed use); PEP (Physical, engineering or chemical
process); BIOL (Biological study); PROC (Process); USES (Uses)
(**supercrit.**; method for producing foodstuffs and/or
feedstuffs by removing undesired lipid constituents using
supercrit. CO2 and by introducing desired lipophilic
substances using compressed CO2)

RN 124-38-9 HCAPLUS

CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)



L79 ANSWER 7 OF 36 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 2001:317898 HCAPLUS

DN 135:200389

ED Entered STN: 04 May 2001

TI Phase equilibria of the vitamins D2, D3 and
K3 in binary systems with CO2 and propane

AU Knez, Z.; Skerget, M.

CS Faculty of Chemistry and Chemical Engineering, University of Maribor,
Maribor, SI-2000, Slovenia

SO Journal of Supercritical Fluids (2001), 20(2), 131-144
CODEN: JSFLEH; ISSN: 0896-8446

PB Elsevier Science B.V.

DT Journal

LA English
 CC 63-8 (Pharmaceuticals)
 Section cross-reference(s): 68
 AB Solubilities of the fat-soluble **vitamins K3, D3 and D2** in **CO2** and propane were measured at temps. 30, 40, 60 and 80°C and over a pressure range of 80-300 bar for **CO2** and 10-110 bar for propane using a static-analytic method. In order to verify weather, the solubility isotherm crosses the three-phase S-L-V line, solid-liquid (S-L) phase transitions of **vitamins K3, D3 and D2** under the pressure of gas were determined with modified capillary method. For all **vitamins** under the pressure of **CO2**, propane and di-Me ether, a neg. dP/dT slope of the S-L-V curve was found. For K3 under the pressure of inert gas (argon and nitrogen), the S-L-V-curve had a pos. dP/dT slope. The solubilities of **vitamins D** in dense **CO2** under the conditions investigated were in the range $0.04+10^{-3}$ - $1.45+10^{-3}$ mole fraction. For **vitamin K3** the solubility in **CO2** was higher and was in the range $0.16+10^{-3}$ - $4.07+10^{-3}$ mole fraction. The solubilities of **vitamins D** in propane were approx. up to 10 times higher as in **CO2** and in the range from $0.15+10^{-3}$ to $12.40+10^{-3}$ mole fraction. Opposite, the solubility of **vitamin K3** in propane was lower than in **CO2** and was in the range from $0.03+10^{-3}$ to $2.99+10^{-3}$ mole fraction. The exptl. S-L-V data were correlated using Peng-Robinson equation of state and binary iteration parameters obtained from best fit were used for solubility calcn. The fit of S-L-V lines showed good results in the case of **vitamin K3** and the order of magnitude of the solid solubilities were well represented.

ST calciferol soly **carbon dioxide** propane pressure;
 cholecalciferol soly **carbon dioxide** propane pressure;
 vitamin K3 soly **carbon dioxide** propane pressure

IT **Vitamins**
 RL: PEP (Physical, engineering or chemical process); PRP (Properties); THU (Therapeutic use); BIOL (Biological study); PROC (Process); USES (Uses)
 (fat-soluble; phase equilibrium of **vitamins D2, D3** and K3 in binary systems with **CO2** and propane)

IT Pressure
 (gas; phase equilibrium of **vitamins D2, D3** and K3 in binary systems with **CO2** and propane)

IT Binary systems
 Phase transition
 Solid solubility
 (phase equilibrium of **vitamins D2, D3** and K3 in binary systems with **CO2** and propane)

IT Extraction
 (**supercrit.**; phase equilibrium of **vitamins D2, D3** and K3 in binary systems with **CO2** and propane for high pressure separation)

IT 50-14-6, **vitamin D2** 58-27-5, **vitamin K3** 67-97-0, **vitamin D3**
 RL: PEP (Physical, engineering or chemical process); PRP (Properties); THU (Therapeutic use); BIOL (Biological study); PROC (Process); USES (Uses)
 (phase equilibrium of **vitamins D2, D3** and K3 in binary systems with **CO2** and propane)

IT 74-98-6, Propane, properties 124-38-9, **Carbon dioxide**, properties
 RL: PRP (Properties)
 (phase equilibrium of **vitamins D2, D3** and K3 in binary systems with **CO2** and propane)

IT 7440-37-1, Argon, properties 7727-37-9, Nitrogen, properties
 RL: PRP (Properties)
 (phase equilibrium of **vitamins D2, D3** and K3 in binary systems with **CO2**, propane, di-Me ether, argon, or

nitrogen)

IT 115-10-6, Dimethylether

RL: PRP (Properties)

(phase equilibrium of **vitamins D2, D3** and K3
in binary systems with CO₂, propane, or di-Me ether)RE.CNT 24 THERE ARE 24 CITED REFERENCES AVAILABLE FOR THIS RECORD
RE

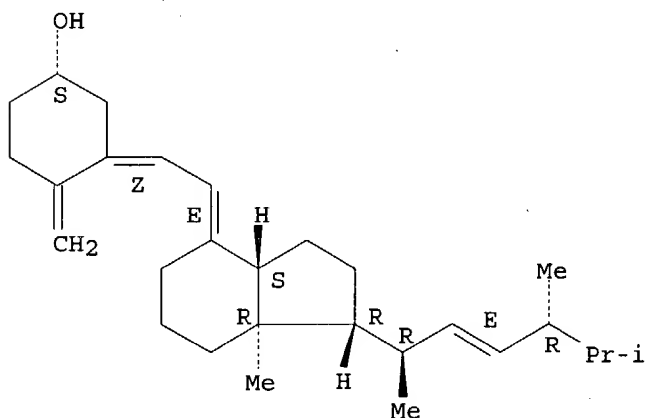
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- (17) Schaffner, D; J Supercrit Fluids 1995, V8, P287 HCAPLUS
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- (22) Tuma, D; Fluid Phase Equilib 1999, V158-160, P743 HCAPLUS
- (23) Weber, W; Proceedings of the Fifth International Symposium on Supercritical Fluids 2000
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IT 50-14-6, **vitamin D2 67-97-0**,**vitamin D3**

RL: PEP (Physical, engineering or chemical process); PRP (Properties); THU (Therapeutic use); BIOL (Biological study); PROC (Process); USES (Uses)

(phase equilibrium of **vitamins D2, D3** and K3in binary systems with CO₂ and propane)

RN 50-14-6 HCAPLUS

CN 9,10-Secoergosta-5,7,10(19),22-tetraen-3-ol, (3 β ,5Z,7E,22E) - (9CI)
(CA INDEX NAME)

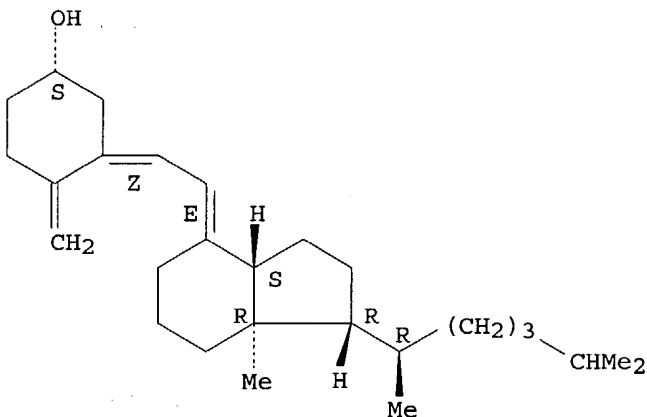
Absolute stereochemistry. Rotation (+).

Double bond geometry as shown.

RN 67-97-0 HCAPLUS

CN 9,10-Secocholesta-5,7,10(19)-trien-3-ol, (3 β ,5Z,7E) - (9CI) (CA INDEX NAME)

Absolute stereochemistry.
Double bond geometry as shown.



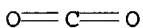
IT 124-38-9, Carbon dioxide, properties

RL: PRP (Properties)

(phase equilibrium of **vitamins D2, D3** and **K3**
in binary systems with **CO2** and propane)

RN 124-38-9 HCAPLUS

CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)



L79 ANSWER 8 OF 36 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 2000:659344 HCAPLUS

DN 134:152447

ED Entered STN: 20 Sep 2000

TI Phase behavior of sterols and vitamins in **supercritical**
CO2

AU Gerszt, R.; Pessoa, F. L. P.; Mendes, M. F.

CS Escola de Quimica (UFRJ), Centro de Tecnologia, Cidade Universitaria, Rio
de Janeiro, CEP 21949-000, Brazil

SO Brazilian Journal of Chemical Engineering (2000), 17(3), 261-270

CODEN: BJCEFZ; ISSN: 0104-6632

PB Associacao Brasileira de Engenharia Quimica

DT Journal

LA English

CC 63-4 (Pharmaceuticals)

AB Extraction with **supercrit.** solvents has been used in different areas, such as petroleum deasphaltation, decaffeination of coffee and tea and in the separation of other types of natural products. The **supercrit.** solvent most frequently utilized in the extraction of natural products is **carbon dioxide (CO2)** due to its several advantages over other solvents such as low cost, non-toxicity and volatility. The design, evaluation and optimization of a **supercrit.** extraction that is based on phase equilibrium require phase equilibrium data. This type of data is very scarce for natural compds. like sterols and vitamins. These natural compds. are produced synthetically, but nowadays interest in their extraction from natural sources is increasing.

Therefore, the objective of this work is to study the thermodyn. modeling equilibrium of systems containing vitamins A, D, E and K, using the predictive

LCVM

model. The sensitivity of critical properties in the calcn. of the phase behavior was also studied. This study proved that the choice of a group contribution method to calculate thermodyn. properties is very important for obtaining good results in the phase equilibrium calcns.

ST

sterol vitamin **supercrit carbon dioxide**

extn; phase behavior sterol vitamin **supercrit** extn

IT

Simulation and Modeling, physicochemical

(phase behavior of sterols and vitamins in **supercrit.**

CO2 extraction)

IT

Sterols

Vitamins

RL: PRP (Properties); PUR (Purification or recovery); PREP (Preparation)

(phase behavior of sterols and vitamins in **supercrit.**

CO2 extraction)

IT

Extraction

(**supercrit.**; phase behavior of sterols and vitamins in

supercrit. **CO2** extraction)

IT

124-38-9, Carbon dioxide, uses

RL: NUU (Other use, unclassified); USES (Uses)

(phase behavior of sterols and vitamins in **supercrit.**

CO2 extraction)

IT

50-14-6P, Vitamin D2 59-02-9P,

α -Tocopherol 67-97-0P, Vitamin D3

119-13-1P, δ -Tocopherol 1406-18-4P, Vitamin E 11103-57-4P,

Vitamin A 11104-38-4P, Vitamin K1

RL: PRP (Properties); PUR (Purification or recovery); PREP (Preparation)

(phase behavior of sterols and **vitamins** in **supercrit**

. CO2 extraction)

RE.CNT 12

THERE ARE 12 CITED REFERENCES AVAILABLE FOR THIS RECORD

RE

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IT

124-38-9, Carbon dioxide, uses

RL: NUU (Other use, unclassified); USES (Uses)

(phase behavior of sterols and vitamins in **supercrit.**

CO2 extraction)

RN

124-38-9 HCAPLUS

CN

Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

O=C=O

IT

50-14-6P, Vitamin D2 67-97-0P,

Vitamin D3

RL: PRP (Properties); PUR (Purification or recovery); PREP (Preparation)

(phase behavior of sterols and **vitamins** in **supercrit**

. CO2 extraction)

RN

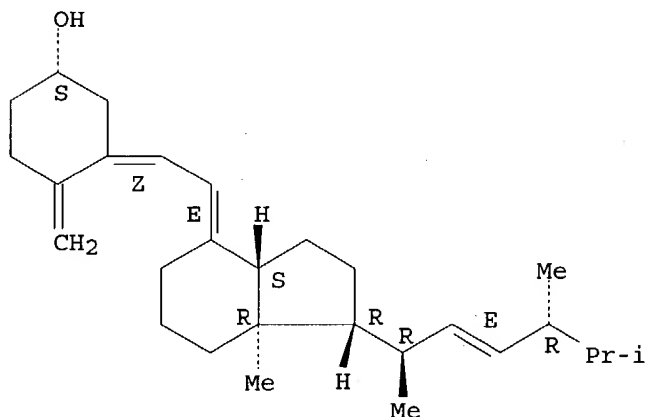
50-14-6 HCAPLUS

CN

9,10-Secoergosta-5,7,10(19),22-tetraen-3-ol, (3 β ,5Z,7E,22E) - (9CI)

(CA INDEX NAME)

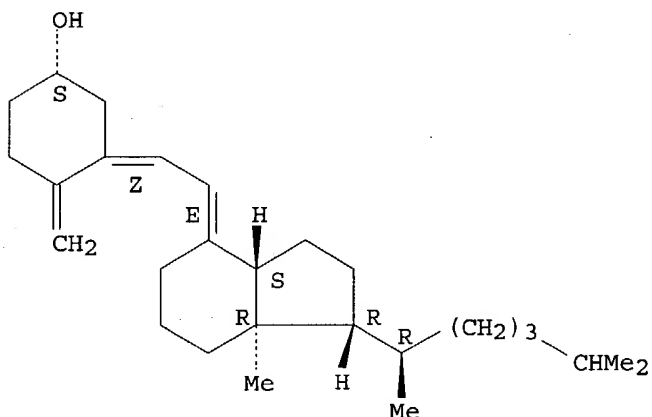
Absolute stereochemistry. Rotation (+).
Double bond geometry as shown.



RN 67-97-0 HCAPLUS

CN 9,10-Secocholesta-5,7,10(19)-trien-3-ol, (3 β ,5Z,7E)-(9CI) (CA INDEX NAME)

Absolute stereochemistry.
Double bond geometry as shown.



L79 ANSWER 9 OF 36 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 2000:436772 HCAPLUS

DN 133:140337

ED Entered STN: 29 Jun 2000

TI Separation of vitamins by **supercritical fluid chromatography** with water-modified **carbon dioxide** as the mobile phase

AU Pyo, D.

CS Department of Chemistry, Kangwon National University, Chuncheon, 200-701, S. Korea

SO Journal of Biochemical and Biophysical Methods (2000), 43(1-3), 113-123

CODEN: JBBMDG; ISSN: 0165-022X

PB Elsevier Science Ireland Ltd.

DT Journal

LA English

CC 64-2 (Pharmaceutical Analysis)

AB **Supercrit. fluid chromatog.** (SFC) has become a technique for solving problems that are difficult to be monitored by other **chromatog.** methods. However, the most widely used fluid, is no more polar than hexane. Polar samples which are difficult to analyze with pure **supercrit. CO2** because of their high polarity can be separated by adding polar modifiers to **supercrit. CO2**. In this study, various vitamins were well separated using water-modified **supercrit. CO2** fluid. The amount of water dissolved in **supercrit. CO2** was measured using an amperometric microsensor made of a thin film of perfluorosulfonate ionomer (PFSI).

ST vitamin sepn **supercrit fluid chromatog; carbon dioxide** mobile phase **chromatog** vitamin sepn

IT **Supercritical fluid chromatography**
(separation of vitamins by **supercrit. fluid chromatog.** with water-modified **carbon dioxide** as mobile phase)

IT Vitamins
RL: ANT (Analyte); ANST (Analytical study)
(separation of vitamins by **supercrit. fluid chromatog.** with water-modified **carbon dioxide** as mobile phase)

IT 50-81-7, Ascorbic acid, analysis 59-43-8, Vitamin B1, analysis 59-67-6, Nicotinic acid, analysis 83-88-5, Vitamin B2, analysis 98-92-0, Nicotinamide **1406-16-2, Vitamin D** 1406-18-4, Vitamin E 8059-24-3, Vitamin B6 12001-79-5, Vitamin K
RL: ANT (Analyte); ANST (Analytical study)
(separation of vitamins by **supercrit. fluid chromatog.** with water-modified **carbon dioxide** as mobile phase)

IT **124-38-9, Carbon dioxide**, analysis
RL: ARU (Analytical role, unclassified); ANST (Analytical study)
(**supercrit.**; separation of vitamins by **supercrit. fluid chromatog.** with water-modified **carbon dioxide** as mobile phase)

RE.CNT 15 THERE ARE 15 CITED REFERENCES AVAILABLE FOR THIS RECORD

RE

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IT **1406-16-2, Vitamin D**
RL: ANT (Analyte); ANST (Analytical study)
(separation of vitamins by **supercrit. fluid chromatog.** with water-modified **carbon dioxide** as mobile phase)

RN 1406-16-2 HCAPLUS

CN Vitamin D (8CI, 9CI) (CA INDEX NAME)

*** STRUCTURE DIAGRAM IS NOT AVAILABLE ***

IT **124-38-9, Carbon dioxide**, analysis
RL: ARU (Analytical role, unclassified); ANST (Analytical study)
(**supercrit.**; separation of vitamins by **supercrit. fluid chromatog.** with water-modified **carbon dioxide** as mobile phase)

RN 124-38-9 HCAPLUS

CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

O=C=O

L79 ANSWER 10 OF 36 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 2000:311007 HCAPLUS
DN 132:339446
ED Entered STN: 14 May 2000
TI Determination of **vitamins D2 and D3** in
pharmaceuticals by **supercritical**-fluid extraction and HPLC
separation with **UV** detection
AU Gamiz-Gracia, L.; Jimenez-Carmona, M. M.; Luque de Castro, M. D.
CS Analytical Chemistry Division, Faculty of Sciences, University of Cordoba,
Cordoba, 14004, Spain
SO Chromatographia (2000), 51(7/8), 428-432
CODEN: CHRGB7; ISSN: 0009-5893
PB Friedrich Vieweg & Sohn Verlagsgesellschaft mbH
DT Journal
LA English
CC 64-2 (Pharmaceutical Analysis)
AB The **supercrit.**-fluid extraction of **vitamins D2**
and **D3** with **carbon dioxide** is reported for
the first time. The extraction recovery was enhanced by direct addition of
di-Et ether to sample contained in the extraction cell. Separation and detection of
the analytes was performed off-line by reversed-phase HPLC with **UV**
-detection. The quantification limit of the method is 4.1 µg for both
analytes, with precision, expressed as relative standard deviation, of 3.8 and
6.3% for **vitamins D2** and **D3**, resp. The
proposed method was applied to the determination of **vitamin D**
in different pharmaceutical products; recoveries were between 85 and 105%.
ST **vitamin D** detn **supercrit** fluid extn; HPLC
vitamin D detn **supercrit** extn; liq
chromatog **vitamin D** detn pharmaceutical
IT **Reversed phase HPLC**
(determination of **vitamins D2** and **D3** in
pharmaceuticals by **supercrit.**-fluid extraction and HPLC with
UV detection)
IT Extraction
(**supercrit.**; determination of **vitamins D2** and
D3 in pharmaceuticals by **supercrit.**-fluid extraction and
HPLC with **UV** detection)
IT 50-14-6, **Vitamin D2** 67-97-0,
Vitamin D3
RL: ANT (Analyte); ANST (Analytical study)
(determination of **vitamins D2** and **D3** in
pharmaceuticals by **supercrit.**-fluid extraction and HPLC with
UV detection)
RE.CNT 24 THERE ARE 24 CITED REFERENCES AVAILABLE FOR THIS RECORD
RE
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(2) Amin, M; J Liquid Chromatogr 1988, V11, P1347 HCAPLUS
(3) Ballesteros, E; Chromatographia 1995, V40, P425 HCAPLUS
(4) Burri, B; J Chromatogr 1997, V762, P201 HCAPLUS
(5) Buskov, S; J Chromatogr 1998, V802, P233 HCAPLUS
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- (19) Savolainen, K; J Pharm Sci 1988, V77, P802 HCAPLUS
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- (22) Schneiderman, M; J Chromatogr 1997, V765, P215 HCAPLUS
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IT 50-14-6, Vitamin D2 67-97-0,

Vitamin D3

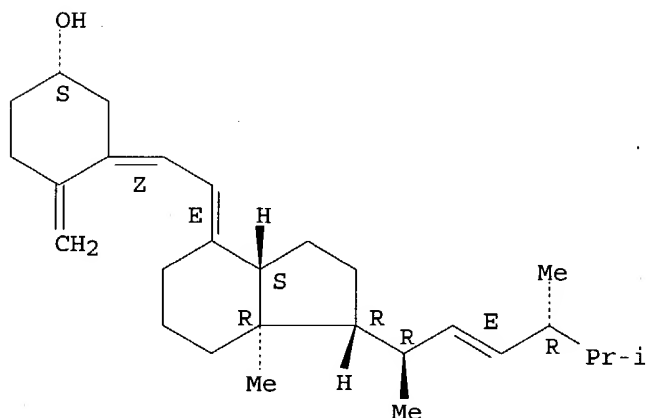
RL: ANT (Analyte); ANST (Analytical study)

(determination of **vitamins D2 and D3** in
pharmaceuticals by **supercrit.**-fluid extraction and HPLC with
UV detection)

RN 50-14-6 HCAPLUS

CN 9,10-Secoergosta-5,7,10(19),22-tetraen-3-ol, (3 β ,5Z,7E,22E)- (9CI)
(CA INDEX NAME)

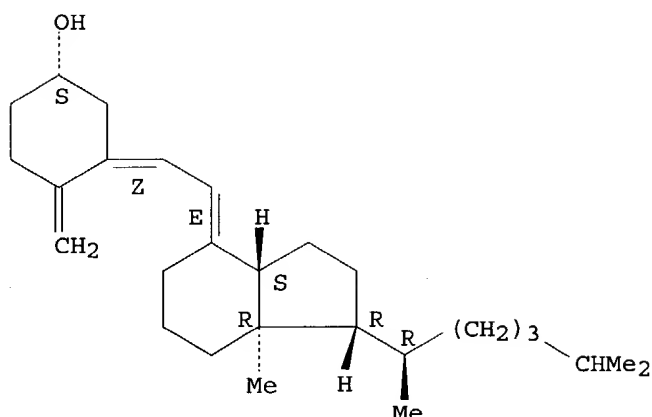
Absolute stereochemistry. Rotation (+).
Double bond geometry as shown.



RN 67-97-0 HCAPLUS

CN 9,10-Secocholesta-5,7,10(19)-trien-3-ol, (3 β ,5Z,7E)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.
Double bond geometry as shown.



L79 ANSWER 11 OF 36 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 2000:12675 HCAPLUS

DN 132:64451

ED Entered STN: 06 Jan 2000

TI Process for the preparation of **vitamin D3** and provitamin D3

IN **Johannsen, Monika**

PA **F. Hoffmann-La Roche A.-G., Switz.**

SO Eur. Pat. Appl., 7 pp.

CODEN: EPXXDW

DT Patent

LA German

IC ICM C07C401-00

CC 32-7 (Steroids)

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	EP 969001	A2	20000105	EP 1999-111617	19990616 <--
	R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO				
	US 2001001801	A1	20010524	US 1999-335022	19990617 <--
	CN 1240209	A	20000105	CN 1999-108675	19990622 <--
	KR 2000006347	A	20000125	KR 1999-23466	19990622 <--
	JP 2000053640	A2	20000222	JP 1999-175755	19990622 <--
	BR 9903274	A	20000516	BR 1999-3274	19990622 <--
PRAI	EP 1998-111490	A	19980623	<--	

AB A process for obtaining **vitamin D3** or **previtamin D3** from a mixture of other steroids, such as dehydrocholesterol, lumisterol and tachysterol, is characterized by separating **vitamin D3** or **previtamin D3** by means of column **chromatog.** with **supercrit.** or liquid **carbon dioxide** as the mobile phase. Thus, **vitamin D3** was separated from a steroid mixture consisting of **vitamin D3**, **previtamin D3**, dehydrocholesterol, lumisterol and tachysterol, using a Hewlett-Packard **chromatograph** (HP G105A SFC) with **supercrit. CO2** (>>31° and >>7.3 MPa) as the mobile phase. Schematics for the separation procedure and a diagram of the **chromatog.** apparatus are presented.

ST column **chromatog** sepn **vitamin D3**
previtamin D3

IT **Liquid chromatography**
Purification
Separation

Supercritical fluid chromatography

(preparation of **vitamin D3** and provitamin D3
via separation from a mixture via column **chromatog.** with
supercrit. or liquid **carbon dioxide**)

IT **Silica gel, uses**

RL: NUU (Other use, unclassified); USES (Uses)
(preparation of **vitamin D3** and provitamin D3
via separation from a mixture via column **chromatog.** with
supercrit. or liquid **carbon dioxide**)

IT **Steroids, preparation**

RL: PUR (Purification or recovery); REM (Removal or disposal); PREP
(Preparation); PROC (Process)
(preparation of **vitamin D3** and provitamin D3
via separation from a mixture via column **chromatog.** with
supercrit. or liquid **carbon dioxide**)

IT **124-38-9, Carbon dioxide, uses**

RL: NUU (Other use, unclassified); USES (Uses)
(preparation of **vitamin D3** and provitamin D3
via separation from a mixture via column **chromatog.** with
supercrit. or liquid **carbon dioxide**)

IT **67-97-0P, Vitamin D3 1173-13-3P,
Previtamin D3**

RL: PUR (Purification or recovery); PREP (Preparation)
(preparation of **vitamin D3** and provitamin D3
via separation from a mixture via column **chromatog.** with
supercrit. or liquid **carbon dioxide**)

IT **115-61-7, Tachysterol 434-16-2, Dehydrocholesterol 474-69-1,
Lumisterol**

RL: REM (Removal or disposal); PROC (Process)
(preparation of **vitamin D3** and provitamin D3
via separation from a mixture via column **chromatog.** with
supercrit. or liquid **carbon dioxide**)

RE.CNT 1 THERE ARE 1 CITED REFERENCES AVAILABLE FOR THIS RECORD

RE

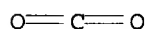
(1) Anon; EP 0969001 A2 HCAPLUS

IT **124-38-9, Carbon dioxide, uses**

RL: NUU (Other use, unclassified); USES (Uses)
(preparation of **vitamin D3** and provitamin D3
via separation from a mixture via column **chromatog.** with
supercrit. or liquid **carbon dioxide**)

RN 124-38-9 HCAPLUS

CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)



IT **67-97-0P, Vitamin D3 1173-13-3P,
Previtamin D3**

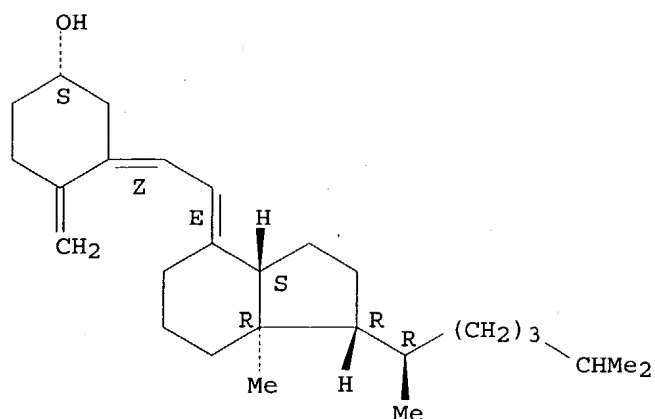
RL: PUR (Purification or recovery); PREP (Preparation)
(preparation of **vitamin D3** and provitamin D3
via separation from a mixture via column **chromatog.** with
supercrit. or liquid **carbon dioxide**)

RN 67-97-0 HCAPLUS

CN 9,10-Secocholesta-5,7,10(19)-trien-3-ol, (3 β ,5Z,7E)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.

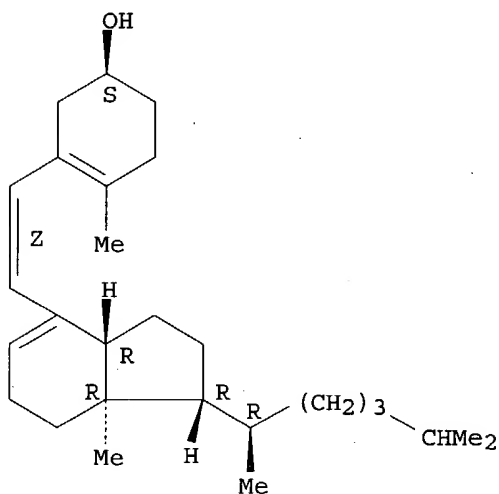
Double bond geometry as shown.



RN 1173-13-3 HCAPLUS

CN 9,10-Secocholesta-5(10),6,8-trien-3-ol, (3 β ,6Z) - (9CI) (CA INDEX NAME)

Absolute stereochemistry.
Double bond geometry as shown.



IT 115-61-7, Tachysterol

RL: REM (Removal or disposal); PROC (Process)

(preparation of **vitamin D3** and provitamin **D3**

via separation from a mixture via column **chromatog.** with **supercrit.** or liquid **carbon dioxide**)

RN 115-61-7 HCAPLUS

CN 9,10-Secoergosta-5(10),6,8,22-tetraen-3-ol, (3 β ,6E,22E) - (9CI) (CA INDEX NAME)

Absolute stereochemistry.
Double bond geometry as shown.

undesired lipid constituents using **supercrit. CO2**
and by introducing desired lipophilic substances using compressed
CO2)

IT Lipids, processes

RL: REM (Removal or disposal); PROC (Process)
(method for producing foodstuffs and/or feedstuffs by removing
undesired lipid constituents using **supercrit. CO2**
and by introducing desired lipophilic substances using compressed
CO2)

IT Breakfast cereal

(musli; method for producing foodstuffs and/or feedstuffs by removing
undesired lipid constituents using **supercrit. CO2**
and by introducing desired lipophilic substances using compressed
CO2)

IT Lipids, biological studies

RL: FFD (Food or feed use); BIOL (Biological study); USES (Uses)
(nonpolar; method for producing foodstuffs and/or feedstuffs by
removing undesired lipid constituents using **supercrit.**
CO2 and by introducing desired lipophilic substances using
compressed **CO2**)

IT Fats and Glyceridic oils, biological studies

RL: FFD (Food or feed use); BIOL (Biological study); USES (Uses)
(oat; method for producing foodstuffs and/or feedstuffs by removing
undesired lipid constituents using **supercrit. CO2**
and by introducing desired lipophilic substances using compressed
CO2)

IT Sterols

RL: FFD (Food or feed use); BIOL (Biological study); USES (Uses)
(phyto-; method for producing foodstuffs and/or feedstuffs by removing
undesired lipid constituents using **supercrit. CO2**
and by introducing desired lipophilic substances using compressed
CO2)

IT Fatty acids, biological studies

RL: FFD (Food or feed use); BIOL (Biological study); USES (Uses)
(polyunsatd., n-3; method for producing foodstuffs and/or feedstuffs by
removing undesired lipid constituents using **supercrit.**
CO2 and by introducing desired lipophilic substances using
compressed **CO2**)

IT Fatty acids, biological studies

RL: FFD (Food or feed use); BIOL (Biological study); USES (Uses)
(polyunsatd., omega-6; method for producing foodstuffs and/or
feedstuffs by removing undesired lipid constituents using
supercrit. CO2 and by introducing desired lipophilic
substances using compressed **CO2**)

IT Fatty acids, biological studies

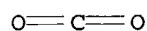
RL: FFD (Food or feed use); BIOL (Biological study); USES (Uses)
(polyunsatd.; method for producing foodstuffs and/or feedstuffs by
removing undesired lipid constituents using **supercrit.**
CO2 and by introducing desired lipophilic substances using
compressed **CO2**)

IT 58-95-7, Tocopherol acetate. 59-02-9, α -Tocopherol 64-17-5D,
Ethanol, polyunsatd. fatty acid esters, biological studies 463-40-1D,
Linolenic acid, esters 506-32-1D, Arachidonic acid, esters
1406-16-2, Vitamin D 1406-18-4, Vitamin E
6217-54-5, Cervonic acid 10417-94-4, Timnodonic acid 11103-57-4,
Provitamin A 57828-26-9D, Lipoic acid, derivs.

RL: FFD (Food or feed use); BIOL (Biological study); USES (Uses)
(method for producing foodstuffs and/or feedstuffs by removing
undesired lipid constituents using **supercrit. CO2**
and by introducing desired lipophilic substances using compressed
CO2)

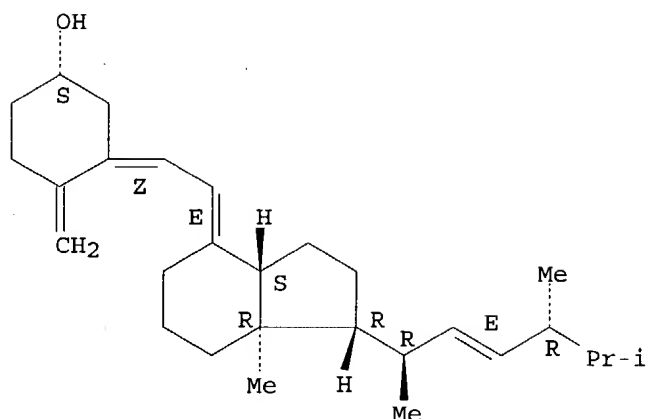
IT 74-98-6, Propane, biological studies

RL: FFD (Food or feed use); PEP (Physical, engineering or chemical



L79 ANSWER 22 OF 36 HCAPLUS COPYRIGHT 2004 ACS on STN
 AN 1961:49155 HCAPLUS
 DN 55:49155
 OREF 55:9533f-h
 ED Entered STN: 22 Apr 2001
 TI Evaluation of **ultraviolet irradiation** by determining
 the photochemical conversion of ergosterol to **vitamin D2**
 AU Jerzykowski, Tadeusz
 CS Inst. Med. Pracy, Zabrze, Pol.
 SO Medycyna Pracy (1960), 11, 331-6
 CODEN: MEPAAX; ISSN: 0465-5893
 DT Journal
 LA Unavailable
 CC 11B (Biological Chemistry: Methods)
 AB A method was developed for relative evaluation of the antirachitic
 activity of **ultraviolet-radiation** sources. A 0.1%
 ergosterol (I) solution in C6H6 under CO2 or N is exposed under
 standardized time and area conditions to the **ultraviolet** light
 to be evaluated. A 0.5-ml. sample of the **irradiated** solution is
 treated with 0.5 ml. 1.4% citraconic anhydride in C6H6, left 30 min. in
 darkness, and evaporated in vacuo to dryness, the residue treated with 3 ml.
 CHCl3 and 2 ml. 1% AcCl in α -dichlorohydrin, let stand for 25 min.,
 and extinction determined photometrically in a 5-cm. thick layer (Eb). Similar
 detns. are run with 0.1% solution of **nonirradiated** I (Ee) and 0.02%
 solution of crystalline **vitamin D2** (Ev). The % conversion =
 100 (Eb - Ee)/(5Ev - Ee).
 IT Electrophoresis, **Electrochromatography**
 (immuno-)
 IT **Ultraviolet** light
 (irradiation by, evaluation of)
 IT 57-87-4, Ergosterol
 (ergocalciferol formation from, in **ultraviolet**
irradiation evaluation)
 IT 50-14-6, Ergocalciferol
 (ergosterol conversion to, in **ultraviolet irradiation**
 evaluation)
 IT 66-02-4, Tyrosine, 3,5-diiodo-
 (formation by thyroid and its determination, Cd effect on)
 IT 50-14-6, Ergocalciferol
 (ergosterol conversion to, in **ultraviolet irradiation**
 evaluation)
 RN 50-14-6 HCAPLUS
 CN 9,10-Secoergosta-5,7,10(19),22-tetraen-3-ol, (3 β ,5Z,7E,22E) - (9CI)
 (CA INDEX NAME)

Absolute stereochemistry. Rotation (+).
 Double bond geometry as shown.



L79 ANSWER 23 OF 36 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 1957:77001 HCAPLUS

DN 51:77001

OREF 51:13891g-i,13892a-i,13893a-c

ED Entered STN: 22 Apr 2001

TI The vitamin D series. XVIII. Partial synthesis of
5,6-trans-vitamin D2

AU Inhoffen, Hans H.; Kath, Joachim F.; Sticherling, Wolfgang; Bruckner,
Klaus

CS Tech. Hochschule, Braunschweig, Germany

SO Ann. (1957), 603, 25-36

DT Journal

LA Unavailable

CC 10 (Organic Chemistry)

GI For diagram(s), see printed CA Issue.

AB cf. C.A. 51, 8121f. All expts. were made under pure N. M.ps. are uncor.

Neutral Number 2 Al2O3 was used in all **chromatograms**, and prepared
either by the Woelm (W.) or the Brockmann (B.) method. Full details are
given for all **chromatographic** sepns. The "Abbau" aldehyde,
C21H34O, m. 56.5-58°, λ 238 m μ (ϵ 20,000) (cf.

Windaus and Riemann, C.A. 37, 59768), (15 g.) in 400 cc. absolute EtOH, 105 g.
4-acetoxycyclohexanone, and 400 cc. 2% EtONa kept 2.5 hrs. at -20°,
poured into H2O, extracted with Et2O, and the extract washed, dried, and
evaporated

gave a brown oil, freed from hydroxycyclohexanone and impurities by
chromatography on Al2O3 (W.) and elution with C6H6, then with
CH2Cl2, yielding 15.3 g. C27 ketone (I, R = H, R' = O) (Ia), which was
acetylated with 168 cc. pyridine and 84 cc. Ac2O, added to N-saturated H2O,
and extracted with Et2O giving I (R = Ac, R' = O) (Ib), isolated by adsorption
on Al2O3 (W.) and eluted with C6H6 (either with or without petr. ether).
On heating impure Ib at 90-100°/5 + 10-3 mm.,
4-acetoxycyclohexanone, identified by its infrared (I.R.) spectrum, distilled
The dry still residue, Ib, yellow oil, decomposed partially on attempted
distillation at 210° in a high vacuum. Ib, dried but not distilled,
 λ 301, ϵ 24,600, **rechromatographed** and saponified at
20°, gave Ia, m. 135° (from aqueous MeOH), λ 300-302
 ϵ 19,800. Another sample of Ib somewhat similarly purified and
saponified gave Ia, m. 138°, λ 300, ϵ 21,300; the mixed
m.ps. showed no depression. Ph3P:CH2 (from 4.1 g. Pn3PMeBr and 1.03 g.
LiPh) treated in Et2O at 20° with constant stirring with 3.9 g. Ib in
500 cc. absolute Et2O, then refluxed 3 hrs., kept overnight, treated with
N-saturated H2O, the Et2O phase removed, the mixture reextd. with Et2O, and all
the exts. washed, dried, and evaporated gave 4.5 g. brown oil which,
chromatographed on Al2O3 (W.), yielded Ph2 (removed by petr.

ether-C₆H₆, 4:1), then 1.59 g. 5,6-**vitamin D2** acetate, I (R = Ac, R' = CH₂) (Ic), nearly colorless oil, λ 272 m μ , ϵ 20,200, whose I.R. spectrum is given and discussed. Ic (prepared from another sample of Ib) showed λ 272-3 m μ , ϵ 19,850. Ic (1.6 g.) in 150 cc. Et₂O saponified with 75 cc. 3% KOH in MeOH 22 hrs. at 20° gave I (R = H, R' = CH₂) (Id), m. 125-6° (preliminary sintering) (from Me₂CO at 0°), λ 272-3 m μ , ϵ 24,600, $[\alpha]_{D20}$ 74.8 \pm 1° (C₆H₆), whose I.R. spectrum in CHCl₃ was determined. Prepared at room temperature from p-PhN₂C₆H₄COCl and Id, I (R = p-PhN₂C₆H₄CO, R' = CH₂) (Ie) formed red crystals, m. 114-17° (preliminary sintering) (from Me₂CO), λ 276-8 and 322 m μ (ϵ 30,000 and 29,700, resp.), $[\alpha]_{D20}$ 25.5 \pm 1.5° (CHCl₃), whose I.R. spectrum is discussed. All spectra are in harmony with the structure assigned to Id, 0.95 g. of which in 74 cc. PhMe and 14 cc. cyclohexanone concentrated to about 60 cc. to insure drying, cooled, and refluxed 45 min. with 0.6 g. (iso-PrO)₃Al, poured into H₂O saturated with N, extracted with Et₂O, and the exts. washed to neutrality, dried, evaporated, and heated 1 hr. at 90°/5.10-3 mm. gave as still residue 853 mg. crude **vitamin D2** ketone (II), yellow oil, λ 263-4 m μ , ϵ 11,100 (Et₂O), 266 m μ , ϵ 10,000 (MeOH); its I.R. spectrum shows the characteristic bands of a dieneone. The semicarbazone of II, m. 209° (from CHCl₃-MeOH), decomposed readily by air and light, λ 292 m μ , ϵ 29,350, $[\alpha]_{D20}$ 24 \pm 1° (CHCl₃), is identical with that isolated by Trippett (C.A. 49, 6286d) from **cis-vitamin D2**. Ia (3.5 g.) in 30 cc. dihydropyran with a drop of POCl₃ kept 2 hrs. at room temperature, shaken with saturated NaHCO₃ solution, extracted repeatedly with Et₂O, the extract dried, evaporated, taken up 3 times in absolute C₆H₆, and each time evaporated to dryness in vacuo, heated at 80°/10-3 mm. to remove impurities, the still residue (4.61 g.) in 30 cc. C₆H₆ filtered through 80 g. Al₂O₃ (B.), and the eluate evaporated gave 4.2 g. stable tetrahydropyranyl ether (III) of Ia (not analyzed), whose I.R. spectrum showed no OH band, λ 302 m μ , ϵ 22,400. Ia (1.5 g.) in CH₂:CHOEt heated 18 hrs. at 125-35° in a sealed tube, **chromatographed** on Al₂O₃ (B.) with petr. ether and petr. ether-C₆H₆ as eluants gave 1.168 g. EtOCHMe ether (IV) of Ia (not analyzed), λ 302 m μ , ϵ 25,000 and 26,800, whose I.R. spectrum indicated etherification. Crystalline **vitamin D2** (V) (source not given) subjected to similar slightly modified etherifications and purifications through Al₂O₃ (B.) gave the following ethers of V: 70% EtOCHMe (VI), colorless oil (not analyzed), λ 266 m μ , ϵ 16,300, and 75% tetrahydropyranyl (VII), C₃₃H₅₂O₂, yellow oil, λ 266 m μ , ϵ 13,200. III (1.31 g.) with Ph₃P:CH₂ (from 0.97 g. Ph₃PMeBr) in absolute Et₂O warmed 90 min. at 60°, kept 15 hrs. at 20°, filtered, washed with Et₂O, and the combined Et₂O exts. washed, dried, evaporated, and **chromatographed** on Al₂O₃ (B.) gave, after removal of Ph₂ and elution with petr. ether-C₆H₆, 28% tetrahydropyranyl ether of Id, colorless oil, λ 272 m μ , ϵ 21,200 or 2,300, whose I.R. spectrum showed the absence of a C=O but the presence of a :CH₂ band. Similarly, IV gave 34% EtOCHMe ether of Id, colorless, λ 272 m μ , ϵ 22,700. IV (0.1 g.) in 3 cc. dioxane refluxed 3 hrs. with 0.6 cc. 1% aqueous (CO₂H)₂, cooled, poured into 100 cc. H₂O, extracted with Et₂O, the exts. washed with aqueous NaHCO₃ and H₂O, dried, evaporated, and the residue taken up in C₆H₆ and **chromatographed** gave 62 mg. Ia. VI, decomposed under varying conditions with (CO₂H)₂, usually gave mixts. of the original ether and V. In 1 experiment 1.6 g. VI in 25 cc. dioxane refluxed 3 hrs. with 10 cc. 1% (CO₂H)₂ gave 1.43 g. oil, which **chromatographed** in petr. ether on Al₂O₃ (B.) and the products eluted with C₆H₆ and then with

Et₂O, yielded 735 mg. isovitamin D₂, pale yellow oil, λ 276 and 286 m μ , highest ϵ 25,200 and 27,200, resp., and 334 mg. V, glassy solid, λ 266 m μ , highest ϵ 18,600. VI in petr. ether shaken with 1% aqueous NaHSO₃ was recovered unchanged. VI (1.3 g.) in 25 cc. absolute EtOH with small amts. of Hg(OAc)₂ heated 18 hrs. in a bomb tube and **chromatographed** gave 0.8 g. V, glass, identified by its I.R. spectrum. VII (0.3 g.) in 15 cc. dioxane treated 18 hrs. at 20° with H₂O (distilled over KOH) and saturated with N and with a gentle stream of CO₂ was recovered unchanged. The tetrahydropyranyl ether of Id (0.16 g.) in 80 cc. warm dioxane refluxed 0.5 hr. with 0.7 cc. 8% (CO₂H)₂ gave a yellow oil, which, even after **chromatography** on Al₂O₃, yielded fractions that were mixts. The EtOCHMe ether of Id (0.45 g.) in 12 cc. dioxane and 2.5 cc. 1% aqueous (CO₂H)₂ kept 130 min. at 90° and **chromatographed** gave 39% iso-vitamin D₂, colorless crystals, λ 287-8 m μ , ϵ ranging from 9900 to 43,000, identified by its **ultraviolet** and I.R. spectra. Id showed only 0.8% of the antirachitic activity manifested by **cis-vitamin D₂**. When Id was **irradiated** with **ultraviolet** light, its activity increased 6-fold. This, however, is not unequivocal evidence that a partial synthesis of **cis-vitamin D₂** had occurred.

- IT Rickets
(antirachitic substances, 5,6-trans-vitamin D and related compds. as)
- IT Infrared spectra
(of **vitamin D** and related compds.)
- IT Benzoic acid, p-phenylazo-, esters with trans-vitamin D₂
Cyclohexanone, 4-(1-ethoxyethoxy)-2-{2-[tetrahydro-7a-methyl-1-(1,4,5-trimethyl-2-hexenyl)-4(3aH)-indanylidene]ethylidene}-
Cyclohexanone, 4-hydroxy-2-{2-[tetrahydro-7a-methyl-1-(1,4,5-trimethyl-2-hexenyl)-4(3aH)-indanylidene]ethylidene}-
Indan, 4-{2-[5-(1-ethoxyethoxy)-2-methyl-enecyclohexylidene]ethylidene}hexahydro-7a-methyl-1-(1,4,5-trimethyl-2-hexenyl)-
- IT Pyran, tetrahydro-2-{4-methylene-3-{2-[tetrahydro-7a-methyl-1-(1,4,5-trimethyl-2-hexenyl)-4(3aH)-indanylidene]ethyl}cyclohexyloxy}-
(stereoisomers)
- IT Acetaldehyde, vinyl acetal polymers
Acetaldehyde, vinyl acetal polymers
(**vitamin D₂**-related acetals)
- IT 1406-16-2, Vitamin D
(-related compds.)
- IT 474-63-5, Ergosta-5,24(28)-dien-3 β -ol
(and esters)
- IT 41043-88-3, Cyclohexanone, 4-hydroxy-, acetate
(formation from 4-hydroxy-2-{2-[tetrahydro-7a-methyl-1-(1,4,5-trimethyl-2-hexenyl)-4(3aH)-indanylidene]ethylidene}cyclohexanone)
- IT 50-14-6, Ergocalciferol
(isomers of, and derivs.)
- IT 51744-67-3, Isovitamin, D₂ 104398-83-6, Cyclohexanone, 4-methylene-3-{2-[tetrahydro-7a-methyl-1-(1,4,5-trimethyl-2-hexenyl)-4(3aH)-indanylidene]ethylidene}-, semicarbazone 108749-29-7, Cyclohexanone, 2-{2-[tetrahydro-7a-methyl-1-(1,4,5-trimethyl-2-hexenyl)-4(3aH)-indanylidene]ethylidene}-4-(tetrahydropyran-2-yloxy)-
115918-16-6, Cyclohexanone, 4-hydroxy-2-{2-[tetrahydro-7a-methyl-1-(1,4,5-trimethyl-2-hexenyl)-4(3aH)-indanylidene]ethylidene}-, acetate
117342-65-1, Cyclohexanone, 4-methylene-3-{2-[tetrahydro-7a-methyl-1-(1,4,5-trimethyl-2-hexenyl)-4(3aH)-indanylidene]ethylidene}-
(preparation of)
- IT 50-14-6, Cyclohexanol, 4-methylene-3-{2-[tetrahydro-7a-methyl-1-(1,4,5-trimethyl-2-hexenyl)-4(3aH)-indanylidene]ethylidene}-
(stereoisomers, and derivs.)
- IT 1406-16-2, Vitamin D
(-related compds.)

RN 1406-16-2 HCAPLUS
 CN Vitamin D (8CI, 9CI) (CA INDEX NAME)

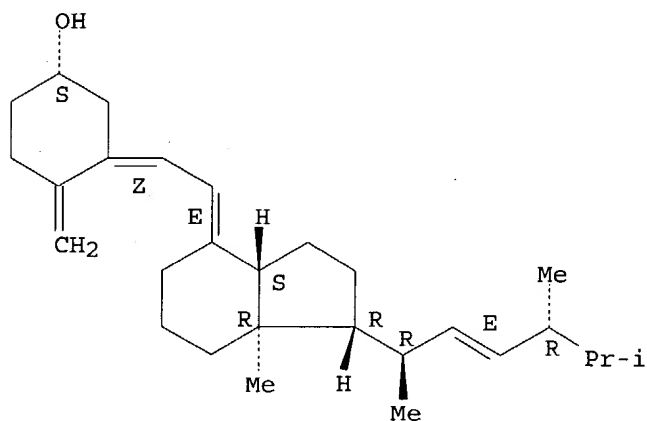
*** STRUCTURE DIAGRAM IS NOT AVAILABLE ***

IT 50-14-6, Ergocalciferol
 (isomers of, and derivs.)

RN 50-14-6 HCAPLUS

CN 9,10-Secoergosta-5,7,10(19),22-tetraen-3-ol, (3 β ,5Z,7E,22E) - (9CI)
 (CA INDEX NAME)

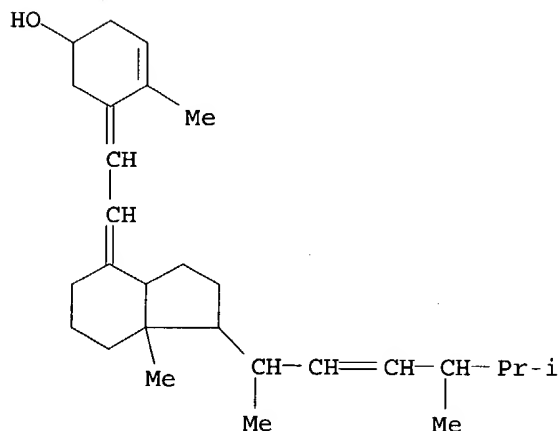
Absolute stereochemistry. Rotation (+).
 Double bond geometry as shown.



IT 51744-67-3, Isovitamin, D2 104398-83-6, Cyclohexanone, 4-methylene-3-{2-[tetrahydro-7a-methyl-1-(1,4,5-trimethyl-2-hexenyl)-4-(3aH)-indanylidene]ethylidene}-, semicarbazone 108749-29-7, Cyclohexanone, 2-{2-[tetrahydro-7a-methyl-1-(1,4,5-trimethyl-2-hexenyl)-4-(3aH)-indanylidene]ethylidene}-4-(tetrahydropyran-2-yloxy)- 115918-16-6, Cyclohexanone, 4-hydroxy-2-{2-[tetrahydro-7a-methyl-1-(1,4,5-trimethyl-2-hexenyl)-4-(3aH)-indanylidene]ethylidene}-, acetate 117342-65-1, Cyclohexanone, 4-methylene-3-{2-[tetrahydro-7a-methyl-1-(1,4,5-trimethyl-2-hexenyl)-4-(3aH)-indanylidene]ethylidene}- (preparation of)

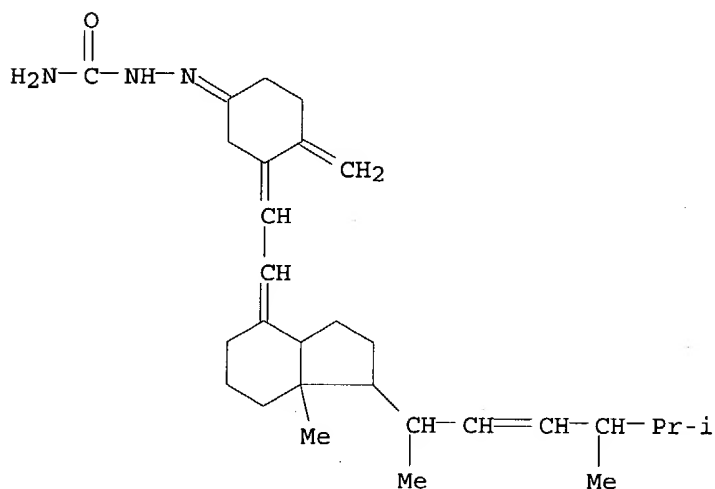
RN 51744-67-3 HCAPLUS

CN 9,10-Secoergosta-1(10),5,7,22-tetraen-3-ol, (3 β ,5E,7E,22E) - (9CI)
 (CA INDEX NAME)



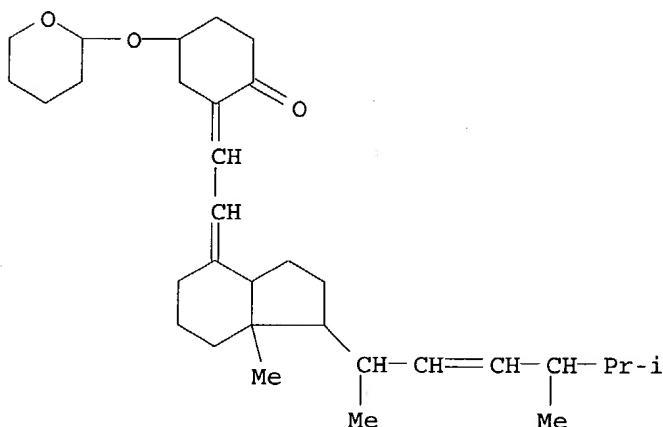
RN 104398-83-6 HCAPLUS

CN Cyclohexanone, 4-methylene-3-[2-[tetrahydro-7a-methyl-1-(1,4,5-trimethyl-2-hexenyl)-4(3aH)-indanylidene]ethylidene]-, semicarbazone (6CI) (CA INDEX NAME)



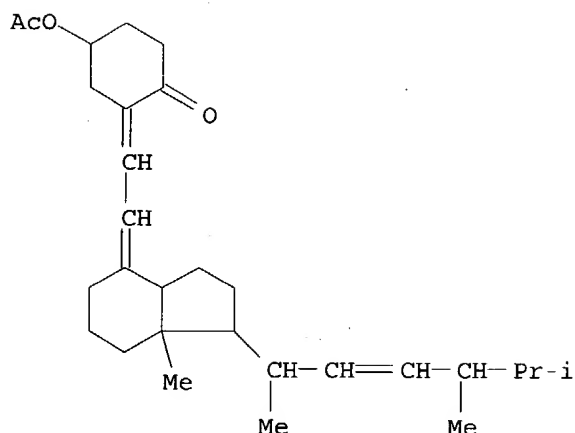
RN 108749-29-7 HCAPLUS

CN Cyclohexanone, 2-[2-[tetrahydro-7a-methyl-1-(1,4,5-trimethyl-2-hexenyl)-4(3aH)-indanylidene]ethylidene]-4-(tetrahydropyran-2-yloxy)- (6CI) (CA INDEX NAME)



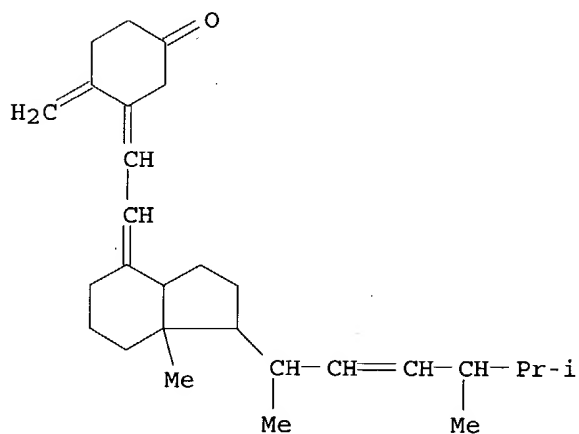
RN 115918-16-6 HCAPLUS

CN Cyclohexanone, 4-hydroxy-2-[2-[tetrahydro-7a-methyl-1-(1,4,5-trimethyl-2-hexenyl)-4(3aH)-indanylidene]ethylidene]-, acetate (6CI) (CA INDEX NAME)



RN 117342-65-1 HCAPLUS

CN Cyclohexanone, 4-methylene-3-[2-[tetrahydro-7a-methyl-1-(1,4,5-trimethyl-2-hexenyl)-4(3aH)-indanylidene]ethylidene]- (6CI) (CA INDEX NAME)

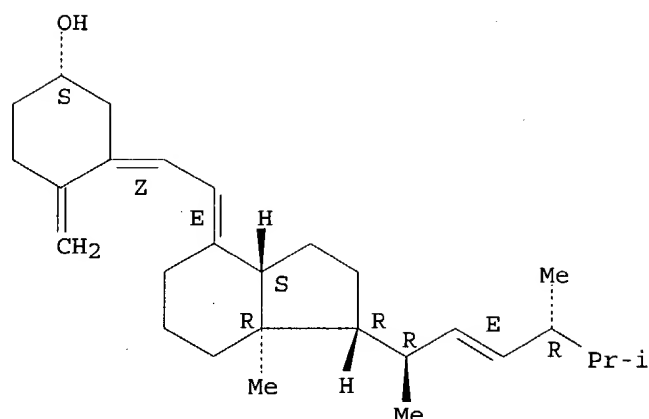


IT 50-14-6, Cyclohexanol, 4-methylene-3-{2-[tetrahydro-7a-methyl-1-(1,4,5-trimethyl-2-hexenyl)-4(3aH)-indanylidene]ethylidene}- (stereoisomers, and derivs.)

RN 50-14-6 HCAPLUS

CN 9,10-Secoergosta-5,7,10(19),22-tetraen-3-ol, (3 β ,5Z,7E,22E)- (9CI) (CA INDEX NAME)

Absolute stereochemistry. Rotation (+).
Double bond geometry as shown.



L79 ANSWER 24 OF 36 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 1956:89264 HCAPLUS

DN 50:89264

OREF 50:16808i,16809a-i,16810a-b

ED Entered STN: 22 Apr 2001

TI **Vitamin D** series. IX. Ring A precursors for syntheses
of tachysterol and its analogs

AU Inhoffen, Hans Herloff; Weissner, Klaus; Quinkert, Gerhard

CS Tech. Hochschule, Braunschweig, Germany

SO Chemische Berichte (1955), 88, 1313-21

CODEN: CHBEAM; ISSN: 0009-2940

DT Journal

LA Unavailable

CC 10 (Organic Chemistry)

OS CASREACT 50:89264

AB cf. C.A. 50, 380e, 8573a. A solution of 4.53 g. 1-methyl-cis-1,2-cyclohexanediol (prepared from 1-methylcyclohexene with KMnO_4) in 5 cc. CCl_4 , 8 cc. CHCl_3 , and 4.4 cc. absolute pyridine treated dropwise below 5° with 3.26 g. tert-BuOCl, kept overnight, extracted with Et₂O, and the Et₂O residue (4.2 g.) in petr. ether **chromatographed** on Al₂O₃ and eluted with petr. ether, then with Et₂O and Et₂O-MeOH (10:1) gave, resp., 0.45 g. tert-BuOH and 1.9 g. 1-hydroxy-1-methyl-2-cyclohexanone (I) (semicarbazone, m. $200-1^\circ$); the column treated with dilute H₂SO₄ and extracted with CHCl_3 gave δ -acetylvaleric acid (semicarbazone, needles from EtOH, m. 143.5°). A stirred solution of 70 g. freshly prepared moist 2-hydroxycyclohexanone in 700 cc. hot H₂O treated with concentrated HCl, then during 5-10 min. with 296 g. $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ in 150 cc. H₂O, cooled to 40° , treated with 220 g. $(\text{NH}_4)_2\text{SO}_4$, and extracted with CH_2Cl_2 gave 1,2-cyclohexanedione, b₂₃ $81-5^\circ$, m. $38-40^\circ$, which gave I with MeMgBr. A solution of 3 g. Li in 800 cc. liquid NH₃ treated with dry C₂H₂ until colorless, then during, and for 2 hrs. after the dropwise addition of 20 g. I in 50 cc. absolute Et₂O, kept 3 hrs., treated with 250 cc. Et₂O and dried NH₄Cl, and evaporated slowly gave 13 g. 1-methyl-2-ethynyl-1,2-cyclohexanediol, b_{2.5} $58-96^\circ$. A solution of 78 g. 4-acetoxycyclohexanone in 500 cc. Et₂O treated dropwise at -5° with 900 cc. MeMgBr solution (from 18 g. Mg), kept at 0° 10 hrs., decomposed (chilling) with the calculated amount dilute H₂SO₄, saturated with NaCl, and extracted with Et₂O gave 34.5 g. 4-acetoxy-1-methylcyclohexanol (II), b_{0.4} $71-3^\circ$; 18 g. KHSO₄ and 30 g. II heated at $90^\circ/0.5$ mm., the product distilled with N, and the distillate extracted with Et₂O and neutralized gave 12.3 g. 4-acetoxy-1-methylcyclohexene (III), b₂₃ 90° . Li (11

g.) in about 1.3 l. liquid NH₃ treated dropwise with stirring with 40 g. p-MeC₆H₄OMe in 60 cc. Et₂O, then after 15 min. with EtOH until colorless; then after evaporating most of the NH₃, with 300 cc. Et₂O and a saturated solution of 95 g. NH₄Cl, the stirring was continued until nearly all the NH₃ was removed, the aqueous solution extracted with addnl. Et₂O, the combined Et₂O solns. washed with saturated NH₄Cl, concentrated to 250 cc., stirred 90 min. with 42 g. (CO₂H)₂ in 550 cc. H₂O, the aqueous layer saturated with NaCl and extracted with addnl. Et₂O, and the combined Et₂O solns. neutralized, dried, and evaporated gave 23.6 g. 1-methyl-1-cyclohexen-4-one [4-methyl-3-cyclohexen-1-one] (IV), b₂₉ 75-84° [semicarbazone, m. 172°; 2,4-dinitrophenylhydrazone, m. 130° (both from EtOH)]. IV (101 g.) in 200 cc. Et₂O added dropwise under N to 11 g. LiAlH₄ in 500 cc. Et₂O, the mixture refluxed 1 hr., decomposed in an ice-salt bath with saturated NH₄Cl solution, and the solid extracted twice with 400 cc. total addnl. Et₂O gave 95.5 g. 1-methyl-1-cyclohexen-4-ol (V), b₃₀₋₁ 90-6° [3,5-dinitrobenzoate, m. 113-13.5° (from EtOH); phenylurethan, m. 108.5-9.5° (from C₆H₆)]. V (39 g.), 58.8 g. freshly-distilled dihydropyran, and a drop POCl₃ shaken with cooling until warm, kept 24 hrs., heated on a water-bath 20 min., cooled, diluted with Et₂O, the solution added to 20% KOH, and the Et₂O residue distilled gave 65.1 g. 4-(2-tetrahydropyranyloxy)-1-methyl-1-cyclohexene (VI), b_{2.5-3} 90-1.5°. Treating 250 cc. 15% NaOH at -10° with 105 cc. chilled 30% H₂O₂, then portionwise (stirring) with 75 g. finely powdered o-C₆H₄-(CO₂)₂O, then with 250 cc. chilled 20% H₂SO₄, extracting the filtrate with Et₂O, washing the exts. with 40% (NH₄)₂SO₄, and drying with Na₂SO₄ gave a perphthalic acid solution (VII). III in 1 volume absolute Et₂O added slowly to an equimolar amount VII, kept 2 days in an icebox, 3 days at room temperature, stirred into chilled aqueous NaOH, the aqueous layer extracted with Et₂O, the combined Et₂O solns. treated with FeSO₄ solution to a neg. KI test, the aqueous layer extracted with Et₂O, and the combined Et₂O solns. washed with a little H₂O, dried (Na₂SO₄), and evaporated gave 62% 4-acetoxy-1-methyl-1,2-oxidocyclohexane (VIII), b₂₄ 113-19°. VI gave 58% 4-(2-tetrahydropyranyloxy)-1-methyl-1,2-oxidocyclohexane, b₅ 116-19°. Refluxing 30 g. VIII with 11 g. KOH in 100 cc. MeOH 30 min., diluting with H₂O, saturating with NaCl, extracting with Et₂O, then during 3 days with CHCl₃, and evaporating the organic solns. gave 11.6 g. 4-hydroxy-1-methyl-1,2-oxidocyclohexane, b₂₆ 126-6.5°. Heating 14 g. VIII and 140 cc. 0.05N HCl on a water-bath 15 min., saturating with NaCl, and extracting 3 days with CHCl₃ gave 12.5 g. 4-acetoxy-1-methyl-trans-1,2-cyclohexanediol, b_{0.02} 126-32°, m. 146-6.5° (from MeOH-hexane). A stirred solution of 39.2 g. VI in 400 cc. EtOH treated at -35° (finally at -15°) with 26 g. KMnO₄ and 29 g. crystalline MgSO₄ in 520 cc. H₂O during 1.5 hrs., filtered from the MnO₂, concentrated in vacuo, saturated with NaCl, extracted with CHCl₃, and the exts. dried and evaporated gave 14.3 g. 4-(2-tetrahydropyranyloxy)-1-methyl-cis-1,2-cyclohexanediol, b_{0.005} 124-37°, m. 101.5°. A solution of 30 g. 1-methyl-2-ethynylcyclohexene (prepared from 1-methyl-2-ethynyl-2-cyclohexanol with POCl₃-pyridine) in 75 cc. absolute Et₂O treated dropwise under N with 520 cc. LiMe solution (1.515 g. active Li), refluxed 30 min., cooled, refluxed 30 min. with 11.1 g. I in 30 cc. Et₂O, cooled, decomposed with chilled saturated NH₄Cl. solution, the crude product (20.6 g., λ 229-30 mμ) in C₆H₆ chromatographed on Al₂O₃, eluted with C₆H₆, C₆H₆-Et₂O (2:1), Et₂O-C₆H₆ (3:1), Et₂O, and Et₂O-MeOH (up to 7%), and the eluates (except with C₆H₆) evaporated gave 7.2 g. α-(1,2-

dihydroxy-2-methylcyclohexenyl)- β -(2-methyl-1-cyclohexenyl)-acetylene (IX), b_{0.01} 130-6°, λ 229-30 m μ (ϵ 13250, MeOH);

1.2 g. IX in 10 cc. absolute Et₂O treated dropwise with 1.6 g. P₂I₄ in 12 cc. CS₂, poured after 10 min. on aqueous NaOH, ice, and Et₂O, shaken with aqueous Na₂S₂O₃, the organic solution evaporated, and the residue (410 mg.) in petr.

ether

filtered through Al₂O₃ gave O-sensitive α,β -bis(2-methyl-1-cyclohexenyl)acetylene, λ 270-2 m μ (ϵ 12280, MeOH).

IT **Chromatography and Adsorption analysis**

(of cyclohexane derivs. (unsatd.))

IT 1,2,4-Cyclohexanetriol, 1-methyl-, trans-, 4-acetate

1,2-Cyclohexanediol, 1-ethynyl-2-methyl-

1,2-Cyclohexanediol, 1-methyl-2-(2-methyl-1-cyclohexen-1-ylethynyl)-

1,2-Cyclohexanediol, 1-methyl-4-(tetrahydropyran-2-yloxy)-, cis-

7-Oxabicyclo[4.1.0]heptan-3-ol, 6-methyl-

7-Oxabicyclo[4.1.0]heptane, 1-methyl-4-(tetrahydropyran-2-yloxy)-

Pyran, 2-(3,4-dihydroxy-4-methylcyclohexyloxy)tetrahydro-

Pyran, tetrahydro-2-(6-methyl-7-oxabicyclo[4.1.0]hept-3-yloxy)-

IT **1406-16-2, Vitamin D**

(-related compds.)

IT 5259-65-4, 3-Cyclohexen-1-one, 4-methyl- 51422-70-9, 3-Cyclohexen-1-ol, 4-methyl-

(and derivs.)

IT **115-61-7, Tachysterol**

(and related compds.)

IT 765-87-7, 1,2-Cyclohexanedione 3476-78-6, Cyclohexanone,

2-hydroxy-2-methyl- 17713-68-7, Heptanoic acid, 6-oxo-, semicarbazone

32591-08-5, Cyclohexene, 1-ethynyl-2-methyl- 52718-65-7,

1,2-Cyclohexanediol, 1-methyl-, cis- 98553-41-4, Cyclohexanone,

2-hydroxy-2-methyl-, semicarbazone 100227-28-9, 7-

Oxabicyclo[4.1.0]heptan-3-ol, 6-methyl-, acetate 100315-12-6, Pyran,

tetrahydro-2-(4-methyl-3-cyclohexen-1-yloxy)- 131974-95-3, Acetylene,

bis(2-methyl-1-cyclohexen-1-yl)- 479075-85-9, 1,4-Cyclohexanediol,

1-methyl-, 4-acetate

(preparation of)

IT **1406-16-2, Vitamin D**

(-related compds.)

RN 1406-16-2 HCAPLUS

CN Vitamin D (8CI, 9CI) (CA INDEX NAME)

*** STRUCTURE DIAGRAM IS NOT AVAILABLE ***

IT **115-61-7, Tachysterol**

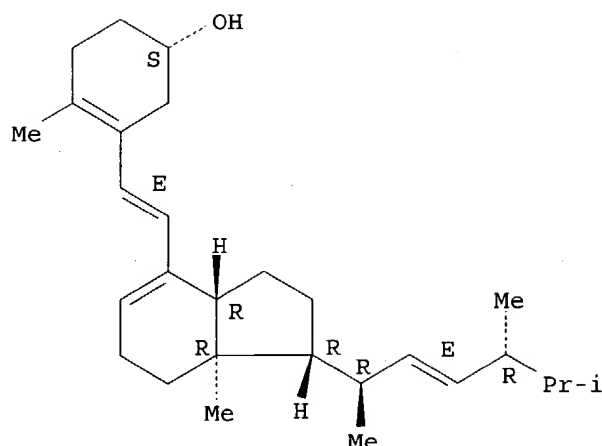
(and related compds.)

RN 115-61-7 HCAPLUS

CN 9,10-Secoergosta-5(10),6,8,22-tetraen-3-ol, (3 β ,6E,22E)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.

Double bond geometry as shown.



L79 ANSWER 25 OF 36 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 1956:57446 HCAPLUS

DN 50:57446

OREF 50:10848b-f

ED Entered STN: 22 Apr 2001

TI Methods for the determinations of fat-soluble **vitamins A, D2, D3, and E**

AU Vendt, V. P.

CS Biochem. Inst., Acad. Sci. Ukr. S.S.R., Kiev

SO Vitaminy, Akad. Nauk Ukr. S.S.R. (1953) 7-29

DT Journal

LA Unavailable

CC 11B (Biological Chemistry: Methods and Apparatus)

AB Detns. of fat-soluble **vitamins A, D2, D3, and E** are

thoroughly discussed and the following methods are considered the most suitable. **Vitamin A**: colorimetric reaction with 1,3-dichloropropan-2-ol (C.A. 41, 2536g) activated by 1-2% HCl added to the reagent; colorimetric, photocolometric, and spectrophotometric methods are described. **Vitamin A** as well as carotene can be determined in the same sample by using wave lengths of 550 and 750 mμ, resp.

Vitamin D2 (calciferol): interference of other sterols is eliminated by digitonin treatment of samples (0.5 ml. 2% aqueous suspension of digitonin per 7 ml. of the solution made up from 25 g. SbCl5 dissolved in 100 ml. dichloroethane), removal of water by addition of anhydrous MgSO4 or NaSO4 to the mixture, filtration, and color formation with the SbCl5 reagent; the colorimetric or spectrophotometric measurements of the color formed is made (within 5-10 min. after the addition of the SbCl5 reagent) at 430 and 530 mμ, and the extinction coefficient of **vitamin**

D2 (ED2) is then calculated by $ED2 = 1.23 E430 - 0.62 E530$. The measurements of the two wave lengths is essential since by

irradiating ergosterol and **chromatography** (through activated Al2O3) the reaction mixture, 4 substances have been isolated which gave the color reaction with the SbCl5 reagent. However, only one addnl. measurement at 530 mμ is required to get the quant. determination of **vitamin D2** in the samples. **Vitamin D3**

is determined by a method similar to that of **vitamin D2**.

Vitamin E: color formation with HNO3 and the photocolometric measurement of the color formed at 470 mμ. To remove various interfering substances from the exptl. samples containing **vitamin E**, **chromatography** (Al2O3) is used, thus giving the following steps for the determination: sampling, saponification of the sample (butter),

extraction of the

unsapond. fraction by Et2O, evaporation of Et2O, solution of the residue in

dichloroethane, chromatographic filtration of the solution under a CO₂ stream, evaporation of dichloroethane, solution of the residue in EtOH, color formation with HNO₃, and spectrophotometric determination of the color formed.

IT Carotene

(determination of)

IT 50-14-6, Vitamin, D2 67-97-0,

Vitamin, D3 1406-18-4, Vitamin E 11103-57-4,
Vitamin, A

(determination or assay of)

IT 50-14-6, Vitamin, D2 67-97-0,

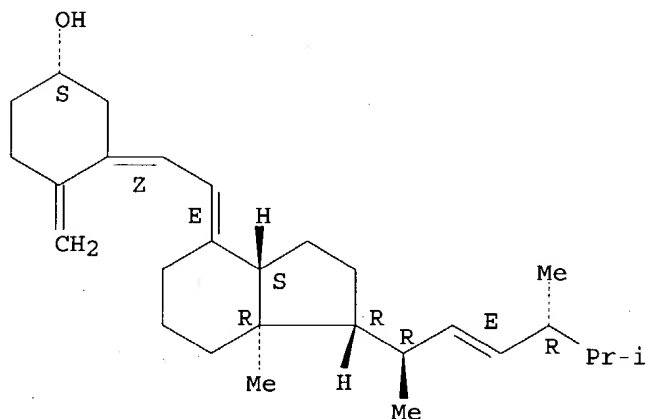
Vitamin, D3

(determination or assay of)

RN 50-14-6 HCAPLUS

CN 9,10-Secoergosta-5,7,10(19),22-tetraen-3-ol, (3 β ,5Z,7E,22E) - (9CI)
(CA INDEX NAME)

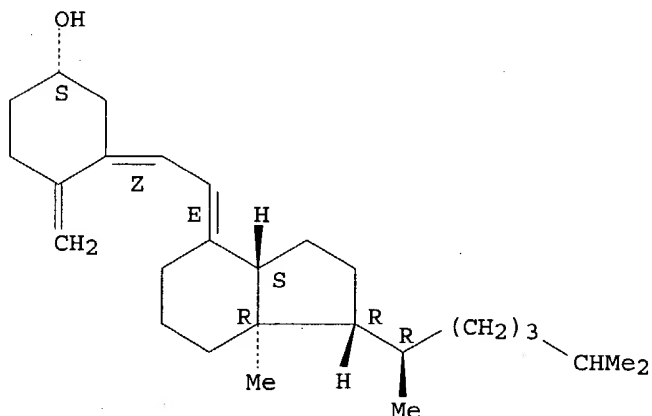
Absolute stereochemistry. Rotation (+).
Double bond geometry as shown.



RN 67-97-0 HCAPLUS

CN 9,10-Secocholesta-5,7,10(19)-trien-3-ol, (3 β ,5Z,7E) - (9CI) (CA INDEX NAME)

Absolute stereochemistry.
Double bond geometry as shown.



L79 ANSWER 26 OF 36 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 1955:49277 HCAPLUS
DN 49:49277

OREF 49:9526b-i,9527a-c

ED Entered STN: 22 Apr 2001

TI The **vitamin D** series. III. Synthesis of a hydrindan derivative by the diene synthesis

AU Inhoffen, Hans Herloff; Kramer, Hans

CS Tech. Hochschule, Braunschweig, Germany

SO Chemische Berichte (1954), 87, 488-96

CODEN: CHBEAM; ISSN: 0009-2940

DT Journal

LA Unavailable

CC 10 (Organic Chemistry)

OS CASREACT 49:49277

GI For diagram(s), see printed CA Issue.

AB cf. C.A. 49, 6291c. The diene reaction between AcOCH:CHCH:CH_2 (I) and 1-methyl-1-cyclopentene-4,5-dione (II) produces an hydrindan derivative in which the AcO and Me groups are ortho to each other. To a solution of 120 g. 2-chlorocyclopentanone in 1200 cc. H_2O heated to 100° 500 g. $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ in 250 cc. H_2O was added dropwise with stirring, the solution cooled to 40° , 360 g. $(\text{NH}_4)_2\text{SO}_4$ added, the solution cooled and extracted with Et_2O or CH_2Cl_2 from which 80.5 g. 1,2-cyclopentanedione (III), b16 $87-8^\circ$, m. $55-6^\circ$, was obtained. III (60 g.) in 250 cc. Et_2O was added to a MeMgBr solution containing 45 g. Mg in about 600 cc. Et_2O , the mixture refluxed 20 min., decomposed with H_2O and dilute H_2SO_4 , continuously extracted with Et_2O for 2 hrs., and the extract fractionated; material up to

b17

78° was removed, and 24.8 g. 1-methyl-1-cyclopenten-5-one (IV), b. $157-8^\circ$, distilled at 760 mm.; in some cases, some 1-methylcyclopentan-1-ol-2-one, b17 $79-81^\circ$, was obtained, which is dehydrated to IV with KHSO_4 . The SeO_2 oxidation product of IV from four 6-g. runs, combined after removal of Se , freed from HOAc at 20 mm., distilled at 12 mm., and recrystd. from Et_2O and petr. ether gave 44% II, m. 84.5° . II (2 g.) in 50 cc. C_6H_6 , 7 cc. pure I, and 15 mg. methylene blue, heated in a bomb 40 hrs. at 120° and filtered, precipitated almost immediately 2.2 g. (55%) 7-acetoxy-8-methyl-5-hydrindene-1,2-dione (V), m. 123.5° (from cyclohexane or Et_2O -petr. ether), λ_{maximum} 259 $\text{m}\mu$ (ϵ 6100, MeOH) and 294 $\text{m}\mu$ ($\text{N}/200 \text{ NaOH}$), violet with FeCl_3 . V (36 mg.) in 20 cc. C_6H_6 was boiled 2 hrs. with 75 mg. $\text{o-C}_6\text{H}_4(\text{NH}_2)_2$ in 10 cc. EtOH and 0.5 cc. HOAc , the solvent evaporated in vacuo, impurities removed by precipitation from Et_2O solution with petr. ether, the solvents evaporated,

and the

residue in 1:1 Et_2O -petr. ether chromatographed on Al_2O_3 ; elution with 80% C_6H_6 in petr. ether separated the quinoxaline derivative of V, λ_{maximum} 240 and 320 $\text{m}\mu$ (ϵ 24800 and 8750, resp.). V (50 mg.) in a little EtOH warmed briefly with several drops of a solution of 1 g. $\text{H}_2\text{NCONHNH}_2 \cdot \text{HCl}$ and 1.5 g. NaOAc in 5 cc. H_2O gave after 30 hrs. the disemicarbazone of V, m. $236-6.5^\circ$. II (2 g.) in 50 cc. C_6H_6 , 5 cc. I, 2.5 cc. Ac_2O , and 15 mg. methylene blue, treated as above, the C_6H_6 removed in vacuo, the residue taken up in Et_2O , filtered over activated C, the solution concentrated, and the product crystallized from cyclohexane gave

2.9 g.

2-enol acetate (VI) of V, m. 73.5° , λ_{maximum} 229-30 $\text{m}\mu$, sometimes obtained as a polymorph, m. 78.5° . VI (100 mg.) in 1.5 cc. EtOH , warmed 30 min. with 1.2 cc. 0.75N K_2CO_3 , acidified with dilute H_2SO_4 , and extracted with Et_2O , gave 60.5 mg. (72%) V. VI (2 g.) in 60 cc. absolute Et_2O added dropwise to 1.6 g. LiAlH_4 in 80 cc. Et_2O , and boiled 1 hr., gave 1.30 g. (93.5%) 1 (or 2), 7-dihydroxy-8-methyl-5-hydrinden-2 (or 1)-one (VII), b10 $125-30^\circ$, which gave a Ag mirror with Tollens reagent. LiAlH_4 reduction of V gave VII. VII (50 mg.) refluxed 30 min. with 2 cc. Ac_2O , poured into H_2O , extracted with Et_2O , and the material from evaporation of the extract warmed with $\text{H}_2\text{NCONHNH}_2$ - NaOAc solution and let stand

hrs., gave the semicarbazone of VII diacetate, m. 217-18° (from EtOAc). Oxidation of 1.5 g. VII in 200 cc. H₂O 48 hrs. at room temperature with 2.9 g. NaIO₄·2H₂O in 200 cc. H₂O and 100 cc. 2N H₂SO₄, addition of 2N NaOH to bring the solution to pH 2-3, extraction with Et₂O and washing with NaHCO₃ and dilute H₂SO₄ under N, and crystallization from a small amount Et₂O gave 1.1 g. (75%) crude cyclohemiacetal lactone, probably VIII, m. 100-5°, which was neutral, gave no semicarbazone, and was colorless with Schiff reagent; on titration VIII consumed 1 mole NaOH, and after treatment with NaHCO₃ VIII gave a rose Schiff test. To a cold solution of 800 mg. VIII in 7.5 cc. Me₂CO 3.75 cc. of a solution containing 100 g. CrO₃ and 160 g. concentrated H₂SO₄/500 cc. was added, the mixture shaken 15 min. at room temperature, 10 cc. H₂O added, CO₂ evolved, the solution extracted with Et₂O, and the extract distilled, giving 400 mg. 2-methyl-3-oxo-4-cyclopentene-1-acetic acid (IX), b₁ 120-30°, m. 98-9.5° after recrystn. from C₆H₆-petr. ether and Et₂O-petr. ether, λ_{maximum} 226 mμ (ε 9600). IX (70 mg.) in 7:3 EtOH-C₆H₆ containing several drops concentrated H₂SO₄ refluxed 4 hrs., part of solvent distilled slowly, the solution diluted with Et₂O, washed with NaHCO₃, and the Et₂O solution evaporated and boiled 2 hrs. with aqueous alc. H₂CONHNH₂ solution gave the semicarbazone of IX Et ester, λ_{maximum} 264 mμ (ε 16000).

IT Synthesis
(diene, of **vitamin D**-related compds.)

IT Spectra
(of **vitamin D**-related compds.)

IT 1-Indanone, 3a,4,7,7a-tetrahydro-2,4(or 2,7)-dihydroxy-7a-methyl-
1-Indanone, 3a,4,7,7a-tetrahydro-2,4(or 2,7)-dihydroxy-7a-methyl-,
semicarbazone diacetate
11H-Indeno[1,2-b]quinoxalin-1-ol, 1,4,4a,11a-tetrahydro-4a-methyl-,
acetate (ester)
11H-Indeno[1,2-b]quinoxalin-4-ol, 1,4,4a,11a-tetrahydro-4a-methyl-,
acetate (ester)
2-Indanone, 3a,4,7,7a-tetrahydro-1,4(or 1,7)-dihydroxy-7a-methyl-,
semicarbazone diacetate
3-Cyclohexene-1-acetic acid, 6-methyl-5-oxo-
3-Cyclohexene-1-acetic acid, 6-methyl-5-oxo-, ethyl ester semicarbazone
Indone, 3a,4,7,7a-tetrahydro-2,4(or 2,7)-dihydroxy-7a-methyl-, diacetate

IT 1,2-Indandione, 3a,4,7,7a-tetrahydro-4(or 7)hydroxy-7a-methyl-
(derivs.)

IT Bromine silver benzoate, (BzO)₂AgBr
(reaction with aromatic compds.)

IT **1406-16-2, Vitamin D**
(-related compds.)

IT 5090-76-6, Cyclohexanepropionic acid, 2-carboxy-2-methyl-6-oxo-
103386-94-3, 1,4(3aH)-Indandione, tetrahydro-7a-methyl-
(and derivs.)

IT 496-10-6, Indan, hexahydro-
(derivs.)

IT 1120-73-6, 2-Cyclopenten-1-one, 2-methyl- 3008-40-0,
1,2-Cyclopentanedione 55767-59-4, Cyclopentanone, 2-hydroxy-2-methyl-
(preparation of)

IT 10130-95-7, 3-Cyclopentene-1,2-dione, 3-methyl-
(reaction with 1,3-butadienyl acetate)

IT 1515-76-0, 1,3-Butadien-1-ol, acetate
(reaction with 3-methyl-3-cyclopentene-1,2-dione)

IT **1406-16-2, Vitamin D**
(-related compds.)

RN 1406-16-2 HCAPLUS

CN Vitamin D (8CI, 9CI) (CA INDEX NAME)

*** STRUCTURE DIAGRAM IS NOT AVAILABLE ***

L79 ANSWER 27 OF 36 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 1955:32479 HCAPLUS

DN 49:32479

OREF 49:6292g-i,6293a-g

ED Entered STN: 22 Apr 2001

TI The **vitamin D** series. II. Partial synthesis of the isotachysterol methyl ether

AU Inhoffen, Hans Herloff; Weissermel, Klaus

CS Tech. Hochschule, Braunschweig, Germany

SO Chemische Berichte (1954), 87, 187-93

CODEN: CHBEAM; ISSN: 0009-2940

DT Journal

LA Unavailable

CC 10 (Organic Chemistry)

OS CASREACT 49:32479

GI For diagram(s), see printed CA Issue.

AB To study the chemical and phys. properties of **vitamin D** the attempt is made to prepare other open-ring steroids with conjugated triene systems. Treating 154 g. 4-methoxycyclohexanone in 200 cc. ether with MeMgBr from 43.5 g. Mg gives 84% 4-methoxy-1-methylcyclohexanol, b₁₉ 106-7°, m. 21.5°, which (72 g.), heated 2 hrs. at 140° with 50 g. KHSO₄, gives 82.5% 4-methoxy-1-methylcyclohexene (I), b₈₀ 91-2°. Treating 20 g. I in 80 cc. CCl₄ with (CH₂CO)₂NBr a few min. at 60-70°, adding 10-20 cc. petr. ether, and keeping the mixture in a refrigerator gives 55% 6-bromo-4-methoxy-1-methylcyclohexene (II), b_{0.03} 60-5°, which (105 g.) is heated in 100 cc. AcOH 2 hrs. at 130-50° with 80 g. anhydrous KOAc, the mixture diluted with H₂O, extracted with ether, and the residue of the ether extract fractionally distilled, giving 3 fractions: (a) 25 g., b₂ 50°, consisting chiefly of I and PhMe, (b) 47 g. 6-acetoxy-4-methoxy-1-methylcyclohexene (III), b₂ 73-83°, and (c) 23 g., b₂ 83-120°, consisting of the starting material and more highly brominated products. Warming 92 g. III in 300 cc. 50% EtOH 3 hrs. at 60° with 31 g. KOH in 500 cc. EtOH and taking up the saponification product in 600 cc. CH₂Cl₂ gives 84.5% 6-hydroxy-4-methoxy-1-methylcyclohexene (IV), b₄ 82-4° (2-anthraquinonecarboxylate m. 170°). Adding slowly 160 g. tert-Bu chromate in 360 cc. anhydrous C₆H₆ to 50 g. IV, keeping the mixture 3 days at 20°, decomposing it in the cold with a saturated (CO₂H)₂ solution until CO₂ is no longer evolved, and extracting the aqueous layer with CH₂Cl₂ gives 69% 6-oxo-4-methoxy-1-methylcyclohexene (V), b₁₀ 90-2°, λ_{maximum} 233 mμ, ε 7,800 (MeOH) (semicarbazone, m. 179.5-80°, λ_{maximum} 263-4 mμ, ε 22,180; 2,4-dinitrophenylhydrazone, red needles, m. 172-3°), also obtained in 70-4% yield from 13 g. IV shaken with 130 g. MnO₂ 70 min. at 20° (phenylsemicarbazone, m. 163°). Passing dry CH:CH into 60 cc. ether and 500 cc. NH₃ containing 4 g. Li until the solution is decolorized, then adding dropwise 17 g. V in 50 cc. ether, keeping the mixture 3 hrs., adding 200 cc. ether and the calculated amount of NH₄Cl, and distilling the residue of the ether solution give 4 fractions:

(d) 6.5 g., b₂ 65°, chiefly o-MeC₆H₄OH (VI), (e) 3.6 g., b₂ 65-75° (VI and traces of 6-hydroxy-4-methoxy-1-methyl-6-ethynylcyclohexene (VII), (f) 2.3 g., b₂ 75-88°, consisting of VII and some VI, and (g) 4.4 g. residue. Redistn. of f gives 9.8% VII, b_{1.5} 84-5.5°, clusters of needles, m. 54.5-5°. Adding dropwise 9.45 cc. MeLi in ether containing 52 mg. Li to 622 mg. VII in 7.5 cc. ether, refluxing the solution 40 min., then adding 1.03 g. C₁₉-ketone (cf. W. and Grundmann, C.A. 30, 8225.6) in 7.5 cc. ether, refluxing the mixture 3 hrs., decomposing it with a saturated NH₄Cl solution, and **chromatographically** fractionating the reaction product (1.65 g.) give 845 mg. of the compound C₂₉H₄₆O₃ (VIII) as a very viscous oil which (2.578 g.) is treated in 15

cc. absolute MeOH with 2.3 g. Pd-C overnight, the solution filtered, the residue washed with 25 cc. MeOH, the filtrate shaken with 400 mg. fresh 5% Pd-C (deactivated with a drop of quinoline) in a H atmospheric, and another 400 mg. poisoned Pd-C added, causing the absorption of 139 cc. H after 275 min. and giving the partially hydrogenated product (IX). Treating 220 mg. IX in 1.5 cc. ether 10 min. with 128 mg. P2I4 in 1.22 cc. CS2, shaking the ether solution with 2N NaOH and a saturated Na2S2O3 solution until colorless, and working up the mixture in a N atmospheric give isotachysterol Me ether (X), which,

after **chromatographic** purification and distillation (b0.001 160-80°), shows λ_{maximum} 280, 290, 302 m μ , ϵ 22,400, 27,200, 20,500; isotachysterol has λ_{maximum} 290 m μ , ϵ 41,000. The reaction mechanism of these condensations is discussed.

IT Spectra

(of isotachysterol methyl ether and related compds.)

IT 2-Cyclohexen-1-ol, 1-{2-[hexahydro-4-hydroxy-7a-methyl-1-(1,4,5-trimethyl-2-hexenyl)-4-indanyl]vinyl}-5-methoxy-2-methyl-

2-Cyclohexen-1-ol, 1-{[hexahydro-4-hydroxy-7a-methyl-1-(1,4,5-trimethyl-2-hexenyl)-4-indanyl]ethynyl}-5-methoxy-2-methyl-

4-Indanol, hexahydro-4-[(1-hydroxy-5-methoxy-2-methyl-2-cyclohexen-1-yl)ethynyl]-7a-methyl-1-(1,4,5-trimethyl-2-hexenyl)-

4-Indanol, hexahydro-4-[2-(1-hydroxy-5-methoxy-2-methyl-2-cyclohexen-1-yl)vinyl]-7a-methyl-1-(1,4,5-trimethyl-2-hexenyl)-

Indan, 5,6,7,7a-tetrahydro-4-[2-(5-methoxy-2-methyl-1-cyclohexen-1-yl)vinyl]-7a-methyl-1-(1,4,5-trimethyl-2-hexenyl)-

Isotachysterol, methyl ether

Semicarbazide, 1-(5-methoxy-2-methyl-2-cyclohexen-1-ylidene)-4-phenyl-

IT 2-Cyclohexen-1-ol, 1-ethynyl-5-methoxy-2-methyl- (and derivs.)

IT 50-14-6, Vitamin, D2 (-related compds.)

IT 98558-30-6, 2-Cyclohexen-1-one, 5-methoxy-2-methyl- (and derivs.)

IT 108420-17-3, 2-Cyclohexen-1-ol, 5-methoxy-2-methyl- (and esters)

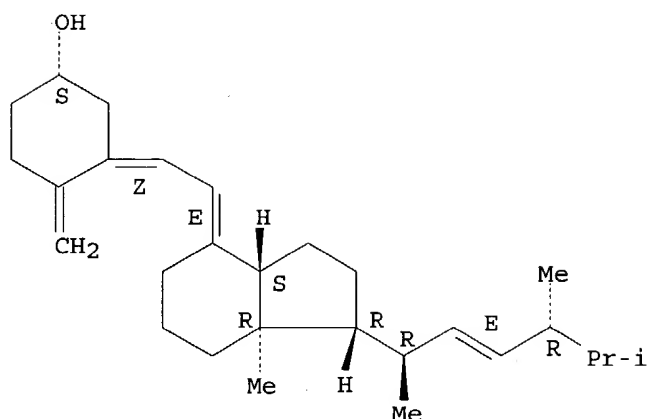
IT 98559-35-4, Ether, 5-bromo-4-methyl-3-cyclohexen-1-yl methyl
102878-81-9, Cyclohexanol, 4-methoxy-1-methyl- 108420-43-5, Ether,
methyl 4-methyl-3-cyclohexen-1-yl 112116-88-8, 2-Anthraquinonecarboxylic
acid, 5-methoxy-2-methyl-2-cyclohexen-1-yl ester
(preparation of)

IT 50-14-6, Vitamin, D2 (-related compds.)

RN 50-14-6 HCAPLUS

CN 9,10-Secoergosta-5,7,10(19),22-tetraen-3-ol, (3 β ,5Z,7E,22E)- (9CI)
(CA INDEX NAME)

Absolute stereochemistry. Rotation (+).
Double bond geometry as shown.



L79 ANSWER 28 OF 36 HCAPLUS COPYRIGHT 2004 ACS on STN
 AN 1953:44659 HCAPLUS
 DN 47:44659
 OREF 47:7531f-i,7532a-i
 ED Entered STN: 22 Apr 2001
 TI Synthesis of **provitamin D** with varied side chains
 AU de Vries, Harmen; Backer, H. J.
 CS Univ. Groningen, Neth.
 SO Recueil des Travaux Chimiques des Pays-Bas et de la Belgique (1952), 71, 719-32
 CODEN: RTCPB4; ISSN: 0370-7539
 DT Journal
 LA French
 CC 10 (Organic Chemistry)
 OS CASREACT 47:44659
 AB cf. C.A. 45, 3857g. 3 β -Hydroxy-17-(1-methylpentyl)- Δ 5,7-androstadiene (7-dehydrobisnorcholesterol) (I), the 17-(1-methylhexyl) homolog (7-dehydronorcholesterol) (II), 17-(1-methylheptyl) homolog (III), 17-(1-methyloctyl) homolog (IV), 17-(1-methyl-5-phenylpentyl) analog (V), and 3 β -hydroxy- Δ 5,7-androstadiene (VI), prepared from the corresponding Δ 5-sterols, were **irradiated** and tested for antirachitic activity; only **irradiated** V showed a little activity. For the preparation of provitamins, all the reactions were carried out under N or CO₂; for bromination and dehydrobromination all compds. and solvents were carefully dried; m.ps. were determined in evacuated capillaries, rotations in 2% CHCl₃ solution free of HCl, absorption spectra in alc. purified with iodine and powdered Zn (3,5-dinitrobenzoates were taken up in CHCl₃ and diluted with alc.). All Δ 5,7-sterols showed absorption maximum at 262.1 (shoulder), 271.3, 281.8, and 293.4 m μ (molar extinctions (ϵ) are given for these 4 λ s in the exptl. details); Δ 4,6-isomers showed absorption maximum in the region 230-48 m μ . Bisnorcholesterol acetate (4.01 g.) in 50 cc. CCl₄ refluxed with 2.14 g. N-bromosuccinimide (VII), **irradiated** ("biosol" lamp) several min. (orange-yellow solution), treated with 0.5 cc. s-collidine, filtered, the filtrate concentrated (20° in vacuo) with several cc. xylene added at the end, the residue taken up in 20 cc. xylene and 10 cc. collidine, heated 15 min. to 140°, concentrated, filtered, the filtrate heated 30 min. with MeOH-KOH, diluted with H₂O, extracted with ether, and the extract washed with dilute H₂SO₄ and H₂O gave 73% I as the 3,5-dinitrobenzoate, m. 160-6° (containing some Δ 4,6-isomer), recrystd. from Me₂CO-MeOH to give the pure compound, m. 193-4° (decomposition), [α]_D -46.5°, ϵ at maximum 15501, 15503, 14275, and 8519, and saponified with MeOH-KOH in C₆H₆ to give pure I, m. 120-20.8°, [α]_D -120°, ϵ at maximum 6576, 9548, 10069, and 5774;

acetate, m. 120.5-1.5°, $[\alpha]$ -79.3°, ϵ at maximum 7181, 10317, 10870, and 6201, resp. Similarly the following were prepared: II [from 10.37 g. norcholesteryl acetate with 5.34 g. VII, isolated in 62% yield as the 3,5-dinitrobenzoate, m. 194.5-5.5° (decomposition) (from $\text{Me}_2\text{CO}-\text{CHCl}_3$), $[\alpha]_D$ -45.4°], m. 124-4.2° (from $\text{Me}_2\text{CO}-\text{MeOH}$; heated in vacuo at 95°), $[\alpha]_D$ -129.2°, ϵ at maximum 7605, 11020, 11640, and 6650; acetate, m. 111.5-12.5° (from $\text{Me}_2\text{CO}-\text{alc.}$), ϵ at maximum 8033, 11424, 11940, and 6815. III [from 8.57 g. 3 β -acetoxy-17-(1-methylheptyl)- Δ^5 -androstene with 4.27 g. VII, isolated in 35% yield as the 3,5-dinitrobenzoate, m. 198-9° (decomposition) (from $\text{CHCl}_3-\text{Me}_2\text{CO}$), $[\alpha]_D$ -40.8°], m. 110.5-11° (meniscus formed at 120°) (from $\text{MeOH}-\text{Me}_2\text{CO}$) (the diene from dilute Me_2CO , dried at 85°, contained 0.5 mol. H_2O), $[\alpha]_D$ -124.5°, ϵ at maximum 7674, 10956, 11562, and 6564; acetate, m. 110.5-11° (from MeOH) (mixed with II gave marked lowering of m.p.), $[\alpha]_D$ -75.6°, ϵ at maximum 8219, 11605, 12215, and 6986. IV, [from 8.854 g. 3 β -acetoxy-17-(1-methyloctyl)- Δ^5 -androstene and 4.272 g. VII, purified via the 3,5-dinitrobenzoate, m. 209-9.5°, in 44% yield], m. 110-10.5° (from dilute Me_2CO) (dried at 85° in vacuo, the crystals contained about 0.33 mol. H_2O), $[\alpha]_D$ -109.7°, ϵ at maximum 6916, 10032, 10566, and 6149; acetate, m. 94-6°, $[\alpha]_D$ -67°, ϵ at maximum 6950, 9726, 10185, and 5846. V, [from 3 β -acetoxy-17-(1-methyl-5-phenylpentyl)- Δ^5 -androstene and VII, isolated as the 3,5-dinitrobenzoate, m. 189-90° (decomposition), $[\alpha]_D$ -34.7°], m. 76-8° (meniscus formed at 112°) (dried in vacuo at 55°, the crystals contained about 0.7 mol. H_2O), $[\alpha]_D$ -66°, ϵ at maximum 8058, 9831, 9925, and 5735. VI, [from 3 β -acetoxy- Δ^5 -androstene (VIII) and VII, isolated in 42% yield as the 3,5-dinitrobenzoate, m. 195.5-6° (decomposition), $[\alpha]_D$ -55°, ϵ at maximum 13527, 12738, 11446, and 7163], m. 157-8°, $[\alpha]_D$ -206.6°, ϵ at maximum 6874, 9816, 10364, and 5886; acetate, m. 118.5-19°, ϵ at maximum 7418, 10144, 10594, and 6098. **Ultraviolet** absorption curves are given graphically. 3-Cholestanone dithioethylene ketal (96% from 3-cholestanone; cf. C.A. 44, 8931g), m. 144-4.6°, with Raney Ni gave cholestane, m. 80°. 3 β -Acetoxy- Δ^5 -androstene-17-one, m. 170.5-1°, with $(\text{CH}_2\text{SH})_2$ and HCl gave 91% dithioethylene ketal (IX), m. 188-9°, $[\alpha]_D$ -88.8°; 4.07 g. IX refluxed 4 hrs. with 25 g. Raney Ni in MeOH gave 85% VIII, m. 95.5-7°. Attempts to make these provitamins by modification of the side chain of ergosterol (X) were unsuccessful because of the difficulties in protecting the conjugated double bond. X (5 g.) kept 24 hrs. at room temperature with 5 cc. Ac_2O and 50 cc. absolute $\text{C}_5\text{H}_5\text{N}$ gave X acetate (XI), m. 179.5° (from $\text{MeOH}-\text{C}_6\text{H}_6$). XI (4.3 g.) heated in a sealed tube 8 hrs. at 135° with 1.6 g. maleic anhydride (XII) in 10 cc. anhydrous xylene (cf. Inhoffen, C.A. 28, 1350.9), concentrated in vacuo, and the solid residue crystallized from ether gave 16% addition product (XIII), m. 217-18° (monoclinic crystals, $\beta = 93.75^\circ$, a:b:c = 3.321:1:1.14), and some needles (sometimes allied with rosettes) (A), m. 174°. Concentration of the ether solution gave a mixture, m. 158-75°, which on crystallization from MeOH yielded needles (B), m. 206-7°, and platelets (C), m. 169-71°; A and B are probably isomers of XIII; C (with an absorption maximum at 251 $\text{m}\mu$) is probably an isomer or a mixture of isomers of XI. Results of addns. of 2.28 millimoles XI and 2.84-9.74 millimoles XII, heated 8 hrs. in various solvents at 135-40°, are tabulated; in the best reaction 20% XIII was obtained with 6.84 millimoles XII in 4 cc. xylene. Dimethylmaleic anhydride, heated at 140° with XI gave no addition product. SO_2 caused isomerization of XI without the formation of sulfones. XI (2.19 g.) in 10 cc. ether, saturated at 0° with SO_2 , heated 5 hrs. in a sealed tube at 100°, the solution washed, concentrated, and the semisolid residue crystallized from Me_2CO , gave 0.9 g.

colorless needles, m. 126-7°, $[\alpha]_D -101.8^\circ$, 0.7 g. solid, m. 125.2-6.2°, and 0.3 g. solid, m. 120°; these products, heated with Ac₂O and separated chromatographically gave several fractions containing no S, probably isomers of XI, m. between 120-6°, which did not depress each other's m.p.; $[\alpha]_D$ varied from -93 to -114°; absorption spectra maximum appeared at 251.5 mμ with a slight shoulder at 245 mμ. The Δ^{7,14}-isomer of XI (0.5 g.) heated 7 hrs. with 1.5 g. XII in 10 cc. C₆H₆, concentrated, the residue saponified with MeOH-KOH, diluted with H₂O, extracted with ether, the ether solution concentrated, and the residue (440 mg., with the same absorption spectrum as before reaction with XII), gave the acetate, m. 127.5°, $[\alpha]_D -93^\circ$, λ_{maximum} 251.3 mμ. The alkaline solution acidified and the product acetylated gave B, m. 205-6°.

IT Rickets
(antirachitic substances, from provitamin D derivs.)

IT Spectra
(of provitamin D derivs.)

IT 3-Cholestanone, cyclic ethylene mercaptole
5,7-Androstadien-3β-ol, 17-(1-methyl-5-phenylpentyl)-
5,7-Androstadien-3β-ol, 17-(1-methyl-5-phenylpentyl)-, 3,5-dinitrobenzoate
5,8-Ethano-15H-cyclopenta[a]phenanthrene-18,19-dicarboxylic anhydride, 1,2,3,4,9,10,11,12,13,14,16,17-dodecahydro-3-hydroxy-10,13-dimethyl-17-(1,4,5-trimethyl-2-hexenyl)-, acetate
5-Androsten-17-one, 3β-hydroxy-, cyclic ethylene mercaptole, acetate
5-Androsten-3β-ol, acetate
7,14,22-Ergostatrien-3-ol, acetate
7,14,22-Ergostatrien-3-ol, maleic anhydride adduct
Cholestane

IT 27-Nor-5,7-cholestadien-3β-ol
5,7-Androstadien-3β-ol
5,7-Androstadien-3β-ol, 17-(1-methylheptyl)-
5,7-Androstadien-3β-ol, 17-(1-methyloctyl)-
5,7-Androstadien-3β-ol, 17-(1-methylpentyl)-
Bisnorcholesterol, 7-dehydro-
Norcholesterol, 7-dehydro-
(and esters)

IT Provitamin, D
(derivs.)

IT Ergosterol, acetate, adduct with maleic anhydride
(isomers)

IT 108-31-6, Maleic anhydride
(adducts, with steroids)

IT 180-20-1, Spiro[3H-cyclopenta[a]phenanthrene-3,2'-[1,3]dithiolane]
187-02-0, Spiro[17H-cyclopenta[a]phenanthrene-17,2'-[1,3]dithiolane]
(derivs.)

L79 ANSWER 29 OF 36 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 1953:33261 HCAPLUS

DN 47:33261

OREF 47:5640a-d

ED Entered STN: 22 Apr 2001

TI Aqueous emulsions of lipide-soluble vitamins

IN Zentner, Margaret R.

PA Hoffmann-La Roche Inc.

DT Patent

LA Unavailable

CC 17 (Pharmaceuticals, Cosmetics, and Perfumes)

FAN.CNT 1

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
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PI US 2628930 19530217 US <--
AB A procedure is described for the preparation of aqueous emulsions of
lipide-soluble
vitamin-active materials (A, D, E) which exhibit a high degree of chemical
and phys. stability. Thus, warm 55 g. of α -tocopherol, 10 g.
glyceryl monostearate (a fatty acid monoester having 12-18 C atoms in the
acid radical), and 5 g. L-ascorbyl palmitate at 80-5° under a
CO₂ atmospheric until a clear molten mass is obtained; add 750 cc. of a
4% gelatin solution (pH 8.5) which contains 30 g. gelatin, 1.35 g. Nipagin
(Me p-hydroxybenzoate), 0.15 g. Nipasol (Pr p-hydroxybenzoate), and 0.375
g. NaOH. After the formation of a buff-colored emulsion add 50 cc. warm
glycerol containing 0.45 g. Nipagin and 0.05 g. Nipasol. Then add 20 cc.
water which contains 0.677 g. citric acid and 4.675 g. Na₂HPO₄. Next add
10 cc. water containing Na₂SO₃. Finally enough water is added to adjust the
volume to 1 l. and enough NaOH to adjust the pH to about 7. Homogenize the
emulsion for 20 min. at 3000 lb. pressure. The homogenized emulsion is
ampuled and sterilized.

IT Vitamins
(aqueous emulsions of fat-soluble)

IT 1406-18-4, Vitamin E
(aqueous emulsions of lipide-soluble)

IT 1406-16-2, Vitamin D
(emulsion containing)

IT 11103-57-4, Vitamin, A
(emulsions (aqueous) of)

IT 1406-16-2, Vitamin D
(emulsion containing)

RN 1406-16-2 HCAPLUS

CN Vitamin D (8CI, 9CI) (CA INDEX NAME)

*** STRUCTURE DIAGRAM IS NOT AVAILABLE ***

L79 ANSWER 30 OF 36 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 1953:32761 HCAPLUS
DN 47:32761
OREF 47:5567c-e
ED Entered STN: 22 Apr 2001
TI Experiences with the chemical determination of **vitamin D**
. III

AU Diemair, W.; Manderscheid-Schwindling, G.
CS Univ. Frankfurt a.M., Germany
SO Zeitschrift fuer Analytische Chemie (1953), 138, 1-8
CODEN: ZANCA8; ISSN: 0372-7920

DT Journal
LA Unavailable
CC 12 (Foods)

AB cf. C.A. 46, 10474e. Impurities in the SbCl₃ reagent or traces of COCl₂
in the CHCl₃ may cause errors by changing the green color toward red.
Vitamin D in irradiated milk must have the
milk fat in an atmospheric of N or CO₂ because it is sensitive to the
atmospheric The **chromatographic** determination is likely to be in error if the
absorption tube is not filled uniformly with Al₂O₃. Sometimes enough
eluant is not used. Care is necessary in separating the layers. From
homogenized milk it is not easy to remove the butter fat. Exptl. data are
given to illustrate these and other sources of error. These data should
prove helpful to those engaged in testing pharmaceutical preps., vitamin
concentrates, ordinary milk, homogenized milk, and baby foods.

IT Milk
(analysis, determination of **vitamin D**)

IT Carcinogenic substances
(food dyes as)

IT **Chromatography and Adsorption analysis**
(of **vitamin D** in milks)

IT Vitamins
(prepns. and concentrates of, vitamin D determination in)
IT Pharmaceuticals
(vitamin D determination in)
IT 1406-16-2, Vitamin D
(determination or assay of)
IT 1406-16-2, Vitamin D
(determination or assay of)
RN 1406-16-2 HCAPLUS
CN Vitamin D (8CI, 9CI) (CA INDEX NAME)

*** STRUCTURE DIAGRAM IS NOT AVAILABLE ***

L79 ANSWER 31 OF 36 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 1952:26697 HCAPLUS
DN 46:26697
OREF 46:4557f-i
ED Entered STN: 22 Apr 2001
TI A new homolog of provitamin D3
AU Strating, J.; Backer, H. J.
SO Proc. Koninkl. Nederland. Acad. Wetenschap. (1951), 54B, 13-15
DT Journal
LA Unavailable
CC 10 (Organic Chemistry)
AB The preparation of 7-dehydro-3-homocholesterol (I) is described. 7-Dehydrocholesteryl chloride (C.A. 45, 3858g) is transformed through its Grignard derivative and reaction with CO₂ into the 3-carboxylic acid (II) (m. 214-15° in vacuo, clear liquid at 260°). II gives with CH₂N₂ the Me ester (III), m. 118.5-19.5° (clear at 127°), [α]_D²⁰ -70.9° (CHCl₃). Reduction of III with LiAlH₄ gives I, m. 125-6° (in vacuo, slight alteration at 120°), [α]_D²⁰ -103.4° (CHCl₃); acetate, m. 97.5-9°, [α]_D¹⁸ -87.2°; benzoate, m. 98-100°, [α]_D¹⁸ -59.9°; 3, 5-dinitrobenzoate, m. 183-4°, [α]_D¹⁸ -24.3°. The ultraviolet spectra of I, II, and III are almost identical with that of 7-dehydrocholesteryl acetate, which proves that no rearrangement of the double bonds has taken place. I gives a precipitate with digitonin, showing that it possesses the β-configuration at C-3. Treatment of the Grignard compound of cholesteryl chloride with CO₂ and esterification of the acid formed with CH₂N₂ gives a Me ester (IV) with a sharp and constant m.p., 101.5-2.5°. Reduction of IV with LiAlH₄ gives 3-homocholesterol (V), m. 129-30°, [α]_D¹⁵ -35.5°, gives a digitonide, and is therefore the 3-β-form. The acetate of V can be transformed by bromination and dehydrobromination into a compound identical with the acetate of I, thus proving the stereochem. structure of the latter. The yields during the various steps of the synthesis of I are nearly quant.

IT Steroids
IT Spectra
(of steroids)
IT 3-Cholesterylcarboxylic acid
3-Cholesterylcarboxylic acid, methyl ester
3-Homocholesterol
3-Homocholesterol, acetate
5,7-Cholestadiene-3β-carboxylic acid
5,7-Cholestadiene-3β-carboxylic acid, methyl ester
5-Cholestene-3β-carboxylic acid
5-Cholestene-3β-methanol
5-Cholestene-3β-methanol, acetate
9(11)-Etioallocholenic acid, 3β-hydroxy-, methyl ester
9(11)-Etiocholenic acid, 3α-hydroxy-, methyl ester
9(11)-Etiocholenic acid, 3α-hydroxy-, methyl ester, acetate
IT 3-Homoprovitamin D3

- (and derivs.)
- IT 3-Homocholesterol, 7-dehydro-
5,7-Cholestadiene-3 β -methanol
(and esters)
- IT 5836-78-2, 5-Cholestene-3 β -carboxylic acid, methyl ester
(preparation of)
- L79 ANSWER 32 OF 36 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 1952:2773 HCAPLUS
DN 46:2773
OREF 46:517d-i,518a-d
ED Entered STN: 22 Apr 2001
TI Compounds related to provitamin D3. III.
Homcholesterol and the corresponding provitamin (3-homoprovitamin D3)
AU Strating, J.; Backer, H. J.
CS Univ. Groningen, Neth.
SO Recueil des Travaux Chimiques des Pays-Bas et de la Belgique (1951
, 70, 389-402
CODEN: RTCPB4; ISSN: 0370-7539
DT Journal
LA English
CC 10 (Organic Chemistry)
AB cf. C.A. 45, 3859a. The term 3-homosterol is proposed for the new type of
homology resulting when the 3-HO group of a sterol is replaced by a
hydroxyalkyl group. The preparation and properties of 3-homocholesterol
(3- β -hydroxymethyl-5-cholestene) (I) and the corresponding
provitamin, 3-homoprovitamin D3 (3 β -hydroxymethyl-5,7-cholestadiene)
(II) are described. Me 5-cholestene-3 β -carboxylate (III), prepared by
esterification of 3-cholesterylcarboxylic acid with CH₂N₂ and separation of the
epimers by recrystn. from MeOH, m. 101.5-2.5°. III with LiAlH₄ in
ether yields 99% I, colorless leaflets from EtOH, m. 129-30°,
[α]_D15 -35.5° (CHCl₃); acetate (IV), colorless leaflets from
EtOH, m. 83-4°, [α]_D20 -25.7° (CHCl₃); benzoate,
leaflets from EtO-Ac, m. 158-9°, [α]_D20 -24.7°
(CHCl₃); 3,5-dinitrobenzoate, yellowish needles from Me₂CO, m.
195.5-6° (in vacuo), [α]_D18 -19.6° (CHCl₃);
digitonide, rosettes of needles. Since pure I could be recovered from the
mother liquor remaining from the digitonide precipitation and also by the
decomposition
of the recrystd. digitonide with anhydrous pyridine, it is concluded that I
consists exclusively of the 3 β -compound IV treated 5 min. with
N-bromosuccinimide in boiling anhydrous CCl₄ yields a complex mixture of
products as a brown viscous mass from which no pure Br compound could be
isolated. Elimination of HBr with anhydrous collidine at 140° in a
CO₂ atmospheric (15 min.), followed by refluxing in 3% alc. KOH in a N
atmospheric (30 min.) yields a brown half-solid residue, which on treatment
with
(O₂N)₂C₆H₃COCl in anhydrous pyridine, and addition of water yields a yellow
precipitate
of crude dinitrobenzoate, m. 184-5° after recrystn. from Me₂CO, and
representing a mixture of 50% of a 5,7- and 4% of a 4,6-cholestadiene (the
remainder probably unchanged IV), as determined by the absorption spectrum.
The yellow crystalline precipitate obtained from the collected filtrates by
dilution with
water was saponified with KOH-MeOH (N atmospheric) and then acetylated with
Ac₂O (
CO₂ atmospheric); after recrystn. from MeOH, the material m. 63°
forms an opaque melt at 71°, and a clear liquid at 80°.
Absorption spectrum studies indicated a mixture of 46% of a 5,7- and 33.5%
of a 4,6-cholestadiene. Further recrystn. from MeOH yields
3 β -acetoxymethyl-5,7-cholestadiene (V), needles, m. 97-7.5°
(in vacuo), [α]_D18.5 -83.8° (CHCl₃). Absorption spectrum
analysis indicated a 2% contamination with a 4,6-cholestadiene. For the

preparation of II an alternate synthesis was chosen. Mg (2.06 g.) and 1.94 g. EtBr in 25 cc. anhydrous peroxide-free ether are heated and mixed prudently (5 hrs.) with 26.16 g. 7-dehydrocholesteryl chloride in 450 cc. ether, the mixture refluxed 20 hrs., concentrated to 175 cc., heated an addnl. 20 hrs.

(all

operations carried out in a N atmospheric), cooled to 0°, a stream of CO₂ led over the mixture 12 hrs., the ether solution decanted from the excess Mg, agitated with 10% H₂SO₄ in a CO₂ atmospheric, the precipitate collected, the residue obtained by evaporation of the filtrate digested with ether, the insol. portion added to the original precipitate, and the entire product recrystd. from C₆H₆, giving 9.64 g. 7-dehydro-3-cholesterylcarboxylic acid (VI), m. 214-15° (opaque melt), 260° (clear liquid) in vacuo. The anomalous m.p. is apparently not a consequence of impurity. VI with CH₂N₂ in ether yields 89% Me ester (VII), long needles from MeOH (CO₂ atmospheric), m. 118.5-19°, clear liquid 127° (in vacuo), [α]_D²⁰ -70.9° (CHCl₃), easily soluble in light petroleum, ether, CHCl₃, C₆H₆, less soluble in Me₂CO, difficultly soluble in MeOH, EtOH. VII, on examination between crossed Nicols, did not show double refraction. VII with LiAlH₄ in anhydrous ether yields a thick white precipitate which, after decomposition of the excess LiAlH₄ with

EtOAc, is

dissolved with dilute H₂SO₄, the ether solution washed with water, dried, evaporated, and the residue recrystd. from MeOH, yielding II as fine white needles, m. 125-6° (slight change at 120°) in vacuo, [α]_D²⁰ -103.4° (CHCl₃); digitonide, rosettes of needles; acetate, fine needles from MeOH, m. 97.5-9° (in vacuo), [α]_D¹⁸ -87.2° (CHCl₃); benzoate, fine needles from MeOH, m. 98-100° (in vacuo), [α]_D¹⁸ -59.9° (CHCl₃); 3,5-dinitrobenzoate, long yellow needles from Me₂CO, m. 183-4° (decomposition) (in vacuo). Direct bromination of III, followed by elimination of HBr, gives a mixture from which no VII could be isolated. The absorption spectra of the compds. discussed, in absolute EtOH from 218-300 mμ, are given in tabular and graphical form. From the absorption curves, it appears that the mol. extinction for II is significantly higher (approx. 1%) than that for 7-dehydrocholesterol. A similar difference is noted in the spectra of the acetates. The phenomenon seems to be of a general character: the mol. extinction appears to increase with increase in mol. weight for a number of steroids with the conjugated system in positions 5-7.

IT Nomenclature

(` ` 3-homosterol'')

IT Spectra

(of steroids)

IT 5,7-Cholestadiene-3β-carboxylic acid

5,7-Cholestadiene-3β-carboxylic acid, methyl ester

IT 3-Homocholesterol

3-Homoprovitamin D₃

5,7-Cholestadiene-3β-methanol

5-Cholestene-3β-methanol

(and derivs.)

IT 3-Homocholesterol, 7-dehydro-

(and esters)

IT Cholestenone-4-C₁₄

(preparation of)

IT 3-Homosterol

(the term)

IT 11025-29-9, Digitonin, compound with cholesterol

(with steroids)

L79 ANSWER 33 OF 36 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 1951:21811 HCAPLUS

DN 45:21811

OREF 45:3859a-f

ED Entered STN: 22 Apr 2001

TI Compounds related to provitamin D3. II. The sulfur analog of provitamin D3

AU Strating, J.; Backer, H. J.

CS Univ., Groningen, Neth.

SO Recueil des Travaux Chimiques des Pays-Bas et de la Belgique (1950), 69, 909-20
CODEN: RTCPB4; ISSN: 0370-7539

DT Journal

LA English

CC 10 (Organic Chemistry)

OS CASREACT 45:21811

AB The synthesis of 5,7-cholestadiene-3-thiol (I) is achieved by the reduction of 7-dehydrocholesteryl thiocyanate (II) or bis(7-dehydro-3-cholesteryl) disulfide (III) with LiAlH_4 . Cholesteryl thiocyanate (IV) is obtained in approx. 30% yield by refluxing 16 g. dry cholesteryl p-toluenesulfonate (V) and 5.6 g. KCNS in 100 cc. anhydrous MeCOEt 12 hrs., filtering the cooled reaction mixture, evaporating the filtrate under reduced pressure, and extracting the residual product several times with small quantities of Me_2CO and crystallizing it from alc. to give pure IV, m. 127-8°. Me_2CO proved to be an unsuitable medium for the above reaction (cf. Muller and B. acte. atyka, C.A. 35, 6243.7), as the presence of traces of water apparently caused the hydrolysis of V with production of di-cholesteryl ether as the only isolable material. Direct reduction of IV with LiAlH_4 in ether affords 81% 5-cholestene-3-thiol (VI), colorless needles from dilute alc., m. 98-9.5°. Preparation of I by the attempted bromination of S-acetyl-5-cholestene-3-thiol (VII), m. 124-5°, $[\alpha]_{20.5D} -52.1^\circ$ (CHCl_3), and the subsequent elimination of HBr proved unsuccessful. A portion of VII reacts with more than 1 Br atom/mol. so that a major portion of VII remains unchanged. Further difficulty was anticipated in the hydrolysis of S-acetyl-7-dehydro-5-cholestene-3-thiol, as VII could not be successfully saponified to the pure thiol VI. Saponification of VII with KOH in MeOH and at room temperature yields dicholesteryl disulfide (VIII) and VI; saponification with a hot (65°) solution of KOH in an evacuated tube yields cholesterol and VI; and saponification with KSH in hot MeOH in an evacuated tube yields VIII and dicholesteryl trisulfide. Crude 7-bromocholesteryl thiocyanate, m. 108-11°, prepared from IV with N-bromosuccinimide, is readily converted to II by heating 20 min. at 140° with anhydrous collidine in a CO_2 atmospheric. The product, after recrystn. from alc., m. 139-140.5° (in vacuo), $[\alpha]_{19.5D} -39.6^\circ$ (CHCl_3). III is obtained by heating II with shaking for 9 hrs. at 90° with NaOEt in an air-free sealed tube; 4 cc. of 2 N HCl and 25 cc. each of C_6H_6 and ether are then added to the mixture, the nonaq. extract washed acid-free with water, dried over Na_2SO_4 , filtered, the solvent removed under reduced pressure, and the residue washed with hot EtOH and recrystd. from AcOEt to yield III, rosettes of needles, m. 161-3°, $[\alpha]_{19D} -12.8^\circ$ (CHCl_3). II with ethereal LiAlH_4 furnishes 67% I; similar reduction of III gives 30% I. I, long needles from EtOH , m. 132-5°, $[\alpha]_{17.5D} -62.2^\circ$ (CHCl_3). The ultraviolet absorption values for the 2 samples of I prepared from II and III are practically identical and compare closely with those for 7-dehydrocholesterol.

IT Spectra
(of 5,7-cholestadien-3 β -ol related compds.)

IT 5,7-Cholestadiene-3-thiol
5-Cholestene-3-thiol
5-Cholestene-3-thiol, acetate
Cholesterol, thiocyanate
Cholesteryl thiocyanate, 7-bromo-
Cholesteryl trisulfide
Disulfide, bis(7-dehydrocholesteryl)

Thiocyanic acid, 5,7-cholestadien-3-yl ester

Thiocyanic acid, 7-bromocholesteryl ester

Thiocyanic acid, cholesteryl esters

IT 2469-23-0; Cholesteryl ether 39879-87-3, Cholesteryl disulfide
(preparation of)

L79 ANSWER 34 OF 36 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 1951:21810 HCAPLUS

DN 45:21810

OREF 45:3858g-i,3859a

ED Entered STN: 22 Apr 2001

TI Compounds related to **provitamin D3**. I.
7-Dehydrocholesteryl chloride and bromide

AU Strating, J.; Backer, H. J.

CS Univ., Groningen, Neth.

SO Recueil des Travaux Chimiques des Pays-Bas et de la Belgique (1950
, 69, 904-8

CODEN: RTCPB4; ISSN: 0370-7539

DT Journal

LA English

CC 10 (Organic Chemistry)

OS CASREACT 45:21810

AB cf. Bide, et al., C.A. 43, 3019i; Bernstein, et al., C.A. 43, 4679a.

7-Dehydrocholesteryl chloride (I) is prepared by the elimination of HBr from 7-bromocholesteryl chloride (II). II is obtained in 37% yield by heating an **irradiated** (Hg-arc lamp) mixture of cholesteryl chloride and N-bromosuccinimide in anhydrous CCl₄ on a water bath for 15 min. The succinimide is filtered off and the filtrate is evaporated in vacuo at room temperature; II, after repeated recrystn. from anhydrous Me₂CO, m. 133.5-4.5° (decomposition). HBr is eliminated by heating II in a CO₂ atmospheric with anhydrous collidine; the temperature of the bath is

raised to

140° over a period of 30 min. and then maintained at that point an addnl. 20 min. The cooled mixture is extracted with peroxide-free ether and with dilute H₂SO₄, the washed and dried ether extract evaporated in vacuo at

room

temperature, and the viscous residue recrystd. from Me₂CO, giving 20% I as fine needles, m. 133.5-4° (in vacuo). 7-Bromocholesteryl chloride (III), m. 135.8-6.2°, and 7-dehydrocholesteryl bromide (IV), m. 147-7.5° (in vacuo), are similarly obtained from cholesteryl bromide in slightly lower yields. The agreement of the absorption spectra of I and IV with that of 7-dehydrocholesterol demonstrates that the synthetic compds. have the same system of conjugated double bonds as is present in **provitamin D**.

IT Spectra

(of 5,7-cholestadien-3 β -ol related compds.)

IT 5-Cholestene, 3 β ,7-dibromo-

5-Cholestene, 7-bromo-3 β -chloro-

L79 ANSWER 35 OF 36 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 1937:53855 HCAPLUS

DN 31:53855

OREF 31:7490b-d

ED Entered STN: 16 Dec 2001

TI Chemical activation of sterols. III. Chemical activation of cholesterol

AU Eck, John C.; Thomas, Byron H.

SO Journal of Biological Chemistry (1937), 119, 621-30

CODEN: JBCHA3; ISSN: 0021-9258

DT Journal

LA Unavailable

CC 11E (Biological Chemistry: Nutrition)

AB cf. C. A. 31, 2264.4. Cholesterol when heated with H₂SO₄, sulfoacetic acid, fuming H₂SO₄, or chlorosulfonic acid in AcOH solution acquires

antirachitic properties. Maximum antirachitic potency was produced by using the proportions of 0.0025 mol. Ac2O, 0.002 mol. concentrated H2SO4 and 0.001 mol. cholesterol in glacial AcOH and heating at 85-90° for 3 hrs.

SO2 and CO2 were evolved in side reactions, the amount of SO2 produced not being correlated with the activity of the product.

Radiation of the product did not affect the potency. Hence the product is not a **provitamin D** activatable by

ultraviolet radiation. Provitamin D

of heated purified cholesterol is not the precursor of the chemically activated product.

IT Sterols

(activation of)

IT Rickets

(antirachitic action, of cholesterol chemically activated)

IT 57-88-5, Cholesterol

(activation of)

L79 ANSWER 36 OF 36 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 1937:10440 HCAPLUS

DN 31:10440

OREF 31:1421f-i,1422a-g

ED Entered STN: 16 Dec 2001

TI Concentration of **vitamin D** from tunafish liver oil

AU Neracher, O.; Reichstein, T.

SO Helvetica Chimica Acta (1936), 19, 1382-91

CODEN: HCACAV; ISSN: 0018-019X

DT Journal

LA Unavailable

CC 10 (Organic Chemistry)

AB The natural **vitamin D** from fish oils is not identical

with calciferol (**vitamin D2**) in that the same amount of

the former, expressed in rat units, has a greater effect on chicks than

the latter (cf. Brockmann, C. A. 30, 6423.1). Accordingly, work on this

problem has been discontinued but details for a simple and reproducible

method of preparing concentrates (20%) containing about 5000 international

units

per mg. are given. Treatment with 3,5-(O2N)2C6H3COCl gave crystalline derivs.

whose parent alcs. proved to be biologically inactive. The saponification of 1

kg. of tunafish oil with 300 g. KOH in 150 cc. air-free H2O and 1.3 l. of

MeOH (distilled over KOH under N2) for 1.5 hrs. under strictly O2-free

conditions, cooling, dilution with 4 l. of N2-saturated H2O and extraction

with Et2O

in a N2 atmospheric, gave 144.5 g. of yellow, partly crystalline unsaponifiable

material (I). When not in process this material, like all other

intermediates, was preserved in ampoules under vacuum. The solution of I in

400 cc. MeOH was cooled in a CO2 atmospheric to 0°. The main

portion of cholesterol crystallized out and gave 48.7 g. of crude oil with a

biol. activity of less than 20 units per mg. The MeOH solution was then

cooled with solid CO2 in a Dewar vessel to -80° and after

30 min. the solid crystalline mass was filtered off through a 3-cm. thick,

finely-powdered, compressed layer of solid CO2 and washed with MeOH

at -80°. The thawed residue was filtered and after vacuum drying

gave 60.0 mg. of a cholesterol fraction (II) as a brown, partially crystalline

oil with a D activity of about 1000 units and a vitamin A content of about

520 units per mg. The MeOH washings yielded 35.2 g. of a dark red-brown

oil with 250 and about 1333 international units of D and A per mg., resp.

Gradual cooling to -15° of a pentane solution of 30 g. II produced 8

g. of white crystals, m. 146°, which showed no A or D color

reactions. A solution of 22 g. II in 200 cc. pentane was run, under

CO2 pressure, through a 60-cm. long, 45-mm. wide

chromatographic column containing 500 g. of washed Al2O3 (Merck,

according to Brockmann). The **chromatogram** was washed with 2 l.

of pentane and yielded 2.608 g. of fluorescent oil with less than 10 units

D per mg. Washing with absolute C₆H₆ and Et₂O (freshly distilled over CaCl₂) gave 5.872 g. of material containing 2000 units D per mg. Elution with a 1:1 mixture of Et₂O and MeOH yielded 11.682 g. of a strongly cholesterol-containing material with about 250 units D per mg. The column retained 1.838 g. of highly colored impurities. The above C₆H₆-Et₂O and Et₂O-MeOH eluates were separated by C₆H₄(CO)₂O according to Ender's method (C. A. 27,523) into 12.7 g. of pentane-soluble alc., 0.569 g. of pentane-insol. alc. and 3.67 of inactive alc.-free material. The 12.7 g. was treated with 30 g. digitonin in 2 l. of 90% alc. and produced 8.48 g. of cholesterol-free alc. which was **chromatographed**. Elution gave a C₆H₆ filtrate containing 3.05 g. of material with a D activity of 6000 units per mg. and an Et₂O filtrate containing 3.13 g. with 5000 units per mg. This latter material (3.025 g.) was dissolved in 10 cc. anhydrous pyridine and refluxed with 4 g. of 3,5-(O₂N)₂C₆H₃COCl in 6 cc. absolute C₆H₆ for 5 min. The cooled reaction mixture was precipitated with Et₂O, filtered and washed with Et₂O. After acidification with ice-cold HCl, neutralization, washing, and drying, the Et₂O was distilled off and the residue was taken up in pentane. The solution was **chromatographed** and eluted with a pentane-C₆H₆ mixture with increasing C₆H₆ content to pure C₆H₆, then with C₆H₆-Et₂O, pure Et₂O and finally Et₂O-Me₂CO mixts. Fractions 4-6 of the 12 eluates yielded 500 mg. of flesh-colored needles of dinitrobenzoate I (III), C₃₂H₄₀N₂O₇, m. 202° (corrected). A solution of 50 mg. III in 16 cc. C₆H₆ was reductively saponified by heating for 15 min. with a solution of 200 mg. SnCl₂ (treated

with

50% KOH to slight turbidity) in 10 cc. absolute alc. The diluted reaction mixture

was extracted with Et₂O and yielded 34 mg. of a biologically inactive oily alc. which could be reconverted to the original III. The concentrated mother liquors from III gave, on long standing in pentane, 280 mg. of yellow needles of dinitrobenzoate II, C₂₇H₃₃N₂O₆, m. 181.5-2.5° (corrected), corresponding to an alc., C₂₀H₃₀-2O, which proved to be biologically inactive. The residual mother liquors were purified through the α-C₁₀H₇NH₂ compound (cf. C. A. 21, 37) and yielded a small amount of a dinitrobenzoate III, m. 113°, from which an inactive alc. was obtained. The above alkaline stannite saponification avoids the oxidation

action of

the NO₂ groups in the usual alkaline saponification of the dinitrobenzoates.

Attempts

to purify the concentrates by distillation at 0.0001 mm. at 150° gave 75% distillates. The cholesterol in tunafish liver oil is accompanied by a relatively rich amount of material precipitated with digitonin and showing a strong

absorption band at about 250 mμ which may be provitamin D.

IT Oils

(tunny-liver, vitamin D from)

IT 1406-16-2, Vitamin D

(preparation from tuna-fish-liver oil)

IT 1406-16-2, Vitamin D

(preparation from tuna-fish-liver oil)

RN 1406-16-2 HCAPLUS

CN Vitamin D (8CI, 9CI) (CA INDEX NAME)

*** STRUCTURE DIAGRAM IS NOT AVAILABLE ***

=> => d all hitstr tot

L96 ANSWER 1 OF 9 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 2001:343243 HCAPLUS

DN 135:131453

ED Entered STN: 14 May 2001

TI Inherent possibilities of improving recovery and selectivity using a long solid phase trap in analytical **supercritical** fluid extraction

- AU Eskilsson, Cecilia Sparr; Turner, Charlotta; Esbjornsson, Annelie;
Mathiasson, Lennart
- CS Department of Analytical Chemistry, Lund University, Lund, S-221 00, Swed.
- SO Journal of Separation Science (2001), 24(4), 297-303
CODEN: JSSCCJ
- PB Wiley-VCH Verlag GmbH
- DT Journal
- LA English
- CC 80-4 (Organic Analytical Chemistry)
Section cross-reference(s): 17, 45
- AB Possibilities of improving recovery and selectivity in **supercrit**
. fluid extraction systems based on solid phase collection by using a longer
trap (5 + 200 mm) instead of a standard trap (5 + 55 mm) were
studied. Such a long trap may give higher sample capacity, and filled
with a **chromatog.** trapping material it gives a column with
higher separation efficiency than the standard trap. The system was tested on
thermally labile substances such as fat-soluble vitamins and aromatic amines.
Quant. recoveries of both vitamins and amines were obtained when using the
long trap combined with modifier in the extracting fluid and trapping temps.
below the b.p. of the modifier conditions that usually cause breakthrough
losses of analytes. Also, the vitamins could be eluted from the long trap
well separated from the lipid fraction, while this was not possible using the
standard trap. The possibility of obtaining selectivity between substances of
similar structures, such as aromatic amines, was studied on different types
of trapping material (ODS, NH₂, and CN). Fairly good fractionation was
achieved using CN. This opens up the possibility to decrease the total
anal. time by eluting the samples in fractions.
- ST solid phase long trap **supercrit** fluid extn; vitamin fat soluble
supercrit extn solid phase long trap; arom amine **supercrit**
fluid extn solid phase long trap
- IT Amines, analysis
RL: ANT (Analyte); PEP (Physical, engineering or chemical process); PRP
(Properties); ANST (Analytical study); PROC (Process)
(aromatic, analytes; inherent possibilities of improving recovery and
selectivity using a long solid phase trap in anal. **supercrit.**
fluid extraction)
- IT Leather
(dyed, samples; improving recovery and selectivity using a long solid
phase trap in anal. **supercrit.** fluid extraction for anal. for
aromatic amines in)
- IT Vitamins
RL: ANT (Analyte); PEP (Physical, engineering or chemical process); PRP
(Properties); ANST (Analytical study); PROC (Process)
(fat-soluble, analytes; inherent possibilities of improving recovery and
selectivity using a long solid phase trap in anal. **supercrit.**
fluid extraction)
- IT Fats and Glyceridic oils, analysis
RL: AMX (Analytical matrix); ANST (Analytical study)
(fish-liver, samples; improving recovery and selectivity using a long
solid phase trap in anal. **supercrit.** fluid extraction for anal.
of)
- IT Trapping apparatus
(inherent possibilities of improving recovery and selectivity using a
long solid phase trap in anal. **supercrit.** fluid extraction)
- IT Silica gel, analysis
RL: ARU (Analytical role, unclassified); NUU (Other use, unclassified);
ANST (Analytical study); USES (Uses)
(reaction products, octadecyl-, aminopropyl-, or cyanopropyl-modified,
trapping materials; inherent possibilities of improving recovery and
selectivity using a long solid phase trap in anal. **supercrit.**
fluid extraction)
- IT Rape oil
RL: AMX (Analytical matrix); ANST (Analytical study)

(samples; improving recovery and selectivity using a long solid phase trap in anal. **supercrit.** fluid extraction for anal. of)

IT Extraction
(**supercrit.**; inherent possibilities of improving recovery and selectivity using a long solid phase trap in anal. **supercrit.** fluid extraction)

IT 59-02-9, α -Tocopherol 68-26-8, vitamin A 79-81-2, Retinyl palmitate 91-94-1, 3,3'-Dichlorobenzidine 95-53-4, o-Toluidine, analysis 119-13-1, 8-Tocopherol 119-90-4, 3,3'-Dimethoxybenzidine 148-03-8, β -Tocopherol **1406-16-2**, vitamin D 1406-18-4, vitamin E 7616-22-0, γ -Tocopherol
RL: ANT (Analyte); ANST (Analytical study)
(analyte; improving recovery and selectivity using a long solid phase trap in anal. **supercrit.** fluid extraction for anal. for)

IT 67-56-1, Methanol, analysis 67-63-0, 2-Propanol, analysis
RL: ARU (Analytical role, unclassified); NUU (Other use, unclassified); ANST (Analytical study); USES (Uses)
(mobile phase containing; improving recovery and selectivity using a long solid phase trap in anal. **supercrit.** fluid extraction for anal. for)

RE.CNT 13 THERE ARE 13 CITED REFERENCES AVAILABLE FOR THIS RECORD
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IT **1406-16-2, vitamin D**
RL: ANT (Analyte); ANST (Analytical study)
(analyte; improving recovery and selectivity using a long solid phase trap in anal. **supercrit.** fluid extraction for anal. for)

RN 1406-16-2 HCAPLUS
CN Vitamin D (8CI, 9CI) (CA INDEX NAME)

*** STRUCTURE DIAGRAM IS NOT AVAILABLE ***

L96 ANSWER 2 OF 9 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 1998:352383 HCAPLUS
DN 128:319006
ED Entered STN: 10 Jun 1998
TI Statistical analysis of liquid trapping efficiencies of fat-soluble vitamins following **supercritical** fluid extraction
AU McDaniel, Lori H.; Long, Gary L.; Taylor, Larry T.
CS Virginia Polytechnic Institute, Department Chemistry, Virginia State University, Blacksburg, VA, 24061, USA
SO Journal of High Resolution Chromatography (1998), 21(4), 245-251
CODEN: JHRCE7; ISSN: 0935-6304
PB Huethig GmbH
DT Journal
LA English
CC 9-9 (Biochemical Methods)
Section cross-reference(s): 68
AB A series of 4 complete factorial expts. were performed to determine the major parameters affecting the trapping efficiencies of fat-soluble vitamins after

supercrit. fluid extraction The parameters varied were the collection solvent, extraction flow rate, collection temperature, restrictor temperature, and collection pressurization. The identity of the collection solvent had the most profound effect. The viscosity and surface tension of the collection solvent had great significance.

ST **supercrit** fluid extn vitamin trapping efficiency; vitamin fat soluble **supercrit** fluid extn

IT Vitamins
 RL: PEP (Physical, engineering or chemical process); PRP (Properties); PROC (Process)
 (fat-soluble; liquid trapping efficiencies of fat-soluble vitamins in **supercrit.** fluid extraction studied by statistical anal.)

IT Surface tension
 (liquid trapping efficiencies of fat-soluble vitamins in **supercrit** . fluid extraction in relation to surface tension of collection solvent)

IT Viscosity
 (liquid trapping efficiencies of fat-soluble vitamins in **supercrit** . fluid extraction in relation to viscosity of collection solvent)

IT Solvents
 (organic, collection; liquid trapping efficiencies of fat-soluble vitamins in **supercrit.** fluid extraction in relation to physico-chemical properties of)

IT Extraction
 (**supercrit.**; liquid trapping efficiencies of fat-soluble vitamins in **supercrit.** fluid extraction studied by statistical anal.)

IT 64-17-5, Ethanol, uses 67-63-0, Isopropanol, uses 110-54-3, Hexane, uses 111-70-6, 1-Heptanol
 RL: NUU (Other use, unclassified); PEP (Physical, engineering or chemical process); PROC (Process); USES (Uses)
 (collection solvent; liquid trapping efficiencies of fat-soluble vitamins in **supercrit.** fluid extraction in relation to physico-chemical properties of)

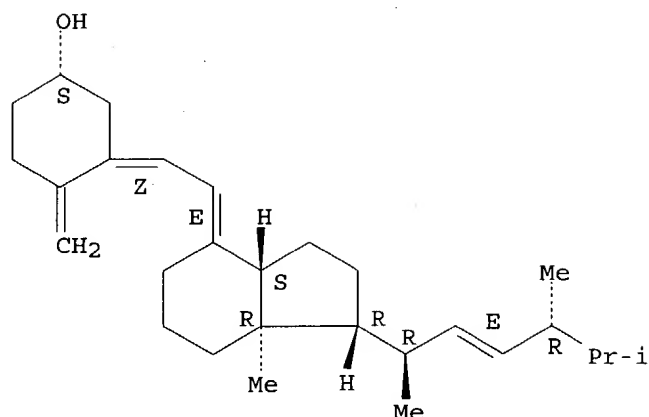
IT 50-14-6, Vitamin d2 59-02-9, α -Tocopherol 67-97-0, Cholecalciferol 68-26-8, all-trans-Retinol 12001-79-5, Vitamin K
 RL: PEP (Physical, engineering or chemical process); PRP (Properties); PROC (Process)
 (liquid trapping efficiencies of fat-soluble vitamins in **supercrit** . fluid extraction studied by statistical anal.)

IT 50-14-6, Vitamin d2 67-97-0, Cholecalciferol
 RL: PEP (Physical, engineering or chemical process); PRP (Properties); PROC (Process)
 (liquid trapping efficiencies of fat-soluble vitamins in **supercrit** . fluid extraction studied by statistical anal.)

RN 50-14-6 HCAPLUS

CN 9,10-Secoergosta-5,7,10(19),22-tetraen-3-ol, (3 β ,5Z,7E,22E) - (9CI)
 (CA INDEX NAME)

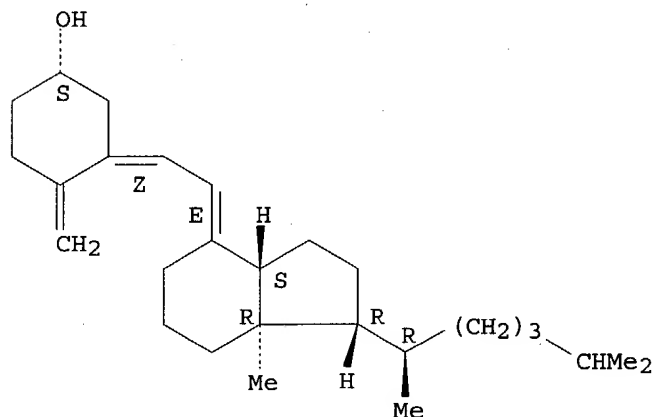
Absolute stereochemistry. Rotation (+).
 Double bond geometry as shown.



RN 67-97-0 HCAPLUS

CN 9,10-Secocholesta-5,7,10(19)-trien-3-ol, (3 β ,5Z,7E)-(9CI) (CA INDEX NAME)

Absolute stereochemistry.
Double bond geometry as shown.



L96 ANSWER 3 OF 9 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 1995:862751 HCAPLUS

DN 123:358081

ED Entered STN: 18 Oct 1995

TI Online SFE-SFC coupling using micropacked columns

AU Ibanez, Elena; Herraiz, Marta; Reglero, Guillermo

CS Instituto de Fermentaciones Industriales, CSIC, Madrid, 28006, Spain

SO Journal of High Resolution Chromatography (1995), 18(8), 507-9

CODEN: JHRCE7; ISSN: 0935-6304

PB Huethig

DT Journal

LA English

CC 80-2 (Organic Analytical Chemistry)

AB A dynamic online coupled system of **supercrit.** fluid extraction (SFE)and **supercrit.** fluid **chromatog.** (SFC) based on

micropacked columns was developed, and the performance of the SFE-SFC system was studied by extraction and anal. of a standard solution of

lipid-soluble

vitamins (vitamin A, A acetate, D2, D3, E, E

acetate, K1, and K3). The use of the micropacked columns enabled SFE-SFC

coupling by an device operating with only 1 pump and a 6-way switching valve integrated into the SF **chromatog.** Data on the performance of the SFE-SFC system are given, e.g. the retention times were reproducible and low detection limits were obtained.

ST **supercrit fluid chromatog** extn micropacked column

IT **Chromatography, supercritical fluid**

(**supercrit. fluid chromatog.-supercrit.**

fluid extraction coupling with micropacked columns)

IT Extraction

(**supercrit., supercrit. fluid chromatog.-**

supercrit. fluid extraction coupling with micropacked columns)

L96 ANSWER 4 OF 9 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 1995:823830 HCAPLUS

DN 123:226193

ED Entered STN: 30 Sep 1995

TI Optimization of Separation of Fat-Soluble Vitamins by

Supercritical Fluid Chromatography Using Serial

Micropacked Columns

AU Ibanez, Elena; Tabera, Javier; Reglero, Guillermo; Herraiz, Marta

CS Instituto de Fermentaciones Industriales, CSIC, Madrid, 28006, Spain

SO Journal of Agricultural and Food Chemistry (1995), 43(10), 2667-71

CODEN: JAFCAU; ISSN: 0021-8561

PB American Chemical Society

DT Journal

LA English

CC 17-1 (Food and Feed Chemistry)

AB The advantages of using two online coupled micropacked columns in

supercrit. fluid chromatog. for analyzing fat-soluble

vitamins are evaluated. A rotatable central composite exptl. design is

used to optimize the combination of some exptl. variables (temperature gradient and pressure gradient) involved in the **chromatog.** separation

Relative standard deviations obtained under the conditions of the exptl.

design giving the highest response are also included. The anal. of

fat-soluble vitamins occurring in real-life samples (a pharmaceutical

preparation

for newborns and an infant formula) is presented.

ST vitamin **supercrit fluid chromatog** infant formula

IT Vitamins

RL: ANT (Analyte); ANST (Analytical study)

(fat-soluble, optimization of separation of fat-soluble vitamins by

supercrit. fluid chromatog. using serial micropacked

columns)

IT Milk substitutes

(human, optimization of separation of fat-soluble vitamins by **supercrit**

. **fluid chromatog.** using serial micropacked columns)

IT 112267-91-1, Protovit

RL: AMX (Analytical matrix); ANST (Analytical study)

(optimization of separation of fat-soluble vitamins by **supercrit.**

fluid chromatog. using serial micropacked columns)

IT 50-14-6, Vitamin D2 58-27-5, Vitamin K3

58-95-7, Vitamin E acetate 67-97-0, Vitamin D3

68-26-8, Vitamin A 1406-18-4, Vitamin E 11104-38-4, Vitamin K1

RL: ANT (Analyte); ANST (Analytical study)

(optimization of separation of fat-soluble vitamins by

supercrit. fluid chromatog. using serial micropacked

columns)

IT 112267-91-1, Protovit

RL: AMX (Analytical matrix); ANST (Analytical study)

(optimization of separation of fat-soluble vitamins by **supercrit.**

fluid chromatog. using serial micropacked columns)

RN 112267-91-1 HCAPLUS

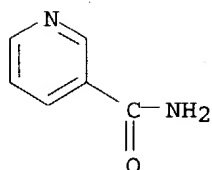
CN L-Ascorbic acid, mixt. with 3-[(4-amino-2-methyl-5-pyrimidinyl)methyl]-5-

(2-hydroxyethyl)-4-methylthiazolium chloride, (R)-2,4-dihydroxy-N-(3-hydroxypropyl)-3,3-dimethylbutanamide, [3aS-(3a α ,4 β ,6a α)]-hexahydro-2-oxo-1H-thieno[3,4-d]imidazole-4-pentanoic acid, 5-hydroxy-6-methyl-3,4-pyridinedimethanol, 3-pyridinecarboxamide, retinol, riboflavin and (3 β ,5Z,7E,22E)-9,10-secoergosta-5,7,10(19),22-tetraen-3-ol (9CI) (CA INDEX NAME)

CM 1

CRN 98-92-0

CMF C6 H6 N2 O

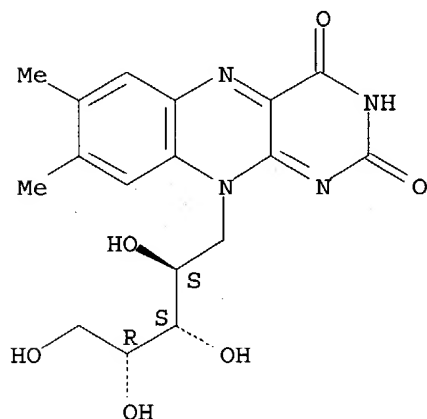


CM 2

CRN 83-88-5

CMF C17 H20 N4 O6

Absolute stereochemistry.

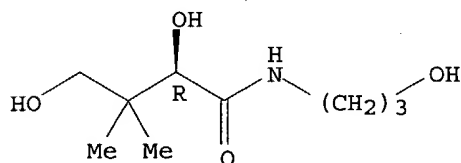


CM 3

CRN 81-13-0

CMF C9 H19 N O4

Absolute stereochemistry.

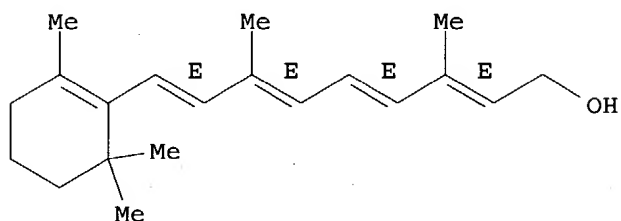


CM 4

CRN 68-26-8

CMF C20 H30 O

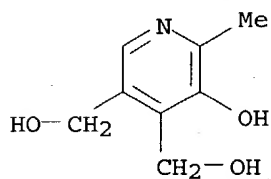
Double bond geometry as shown.



CM 5

CRN 65-23-6

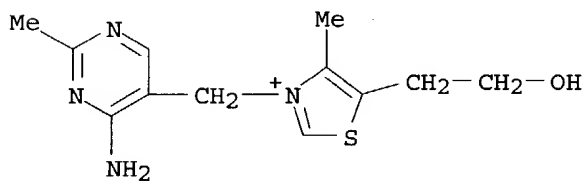
CMF C8 H11 N O3



CM 6

CRN 59-43-8

CMF C12 H17 N4 O S . Cl

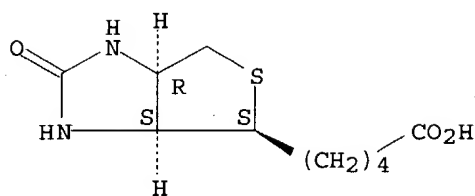
● Cl⁻

CM 7

CRN 58-85-5

CMF C10 H16 N2 O3 S

Absolute stereochemistry. Rotation (+).

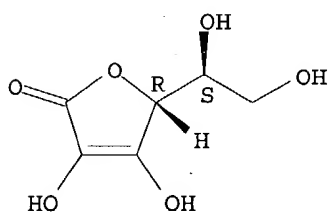


CM 8

CRN 50-81-7

CMF C6 H8 O6

Absolute stereochemistry.

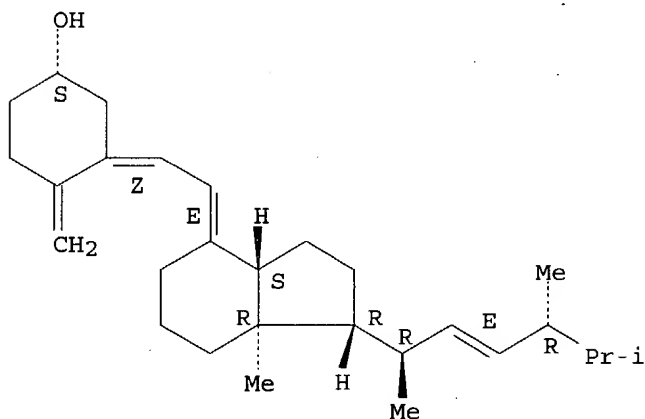


CM 9

CRN 50-14-6

CMF C28 H44 O

Absolute stereochemistry. Rotation (+).
Double bond geometry as shown.



IT 50-14-6, Vitamin D2 67-97-0,

Vitamin D3

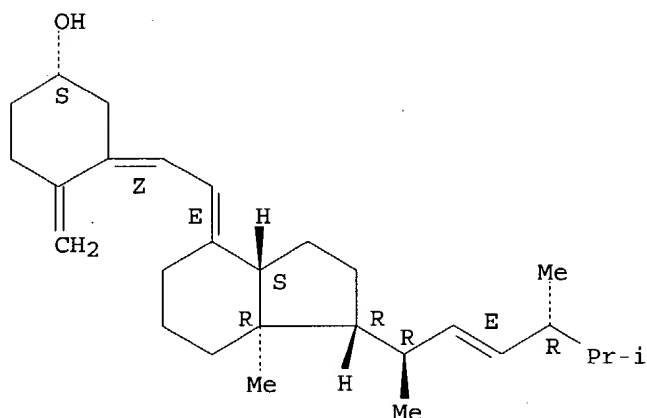
RL: ANT (Analyte); ANST (Analytical study)

(optimization of separation of fat-soluble vitamins by
supercrit. fluid chromatog. using serial micropacked
columns)

RN 50-14-6 HCAPLUS

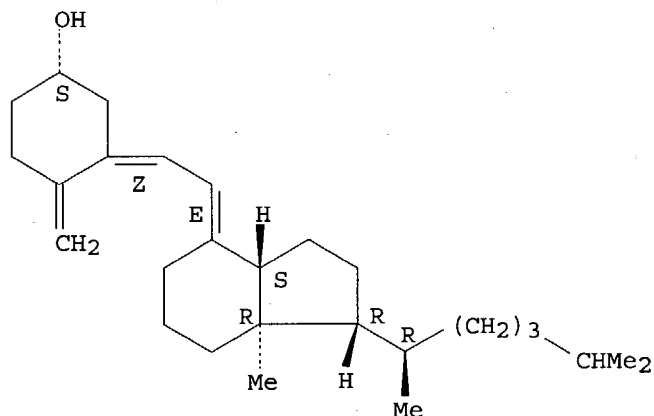
CN 9,10-Secoergosta-5,7,10(19),22-tetraen-3-ol, (3 β ,5Z,7E,22E)- (9CI)
(CA INDEX NAME)

Absolute stereochemistry. Rotation (+).
Double bond geometry as shown.



RN 67-97-0 HCAPLUS
CN 9,10-Secocholesta-5,7,10(19)-trien-3-ol, (3 β ,5Z,7E) - (9CI) (CA INDEX NAME)

Absolute stereochemistry.
Double bond geometry as shown.



L96 ANSWER 5 OF 9 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 1995:549051 HCAPLUS
DN 122:274206
ED Entered STN: 13 May 1995
TI Optimization of fat-soluble vitamin separation by **supercritical**
fluid **chromatography**
AU Ibanez, E.; Tabera, J.; Reglero, G.; Herraiz, M.
CS Inst. Ferment. Ind., CSIC, Madrid, 28006, Spain
SO Chromatographia (1995), 40(7/8), 448-52
CODEN: CHRGB7; ISSN: 0009-5893
PB Vieweg
DT Journal
LA English
CC 64-2 (Pharmaceutical Analysis)
AB Fat-soluble vitamin separation, achievable using a micropacked SFC column
loaded
with CW 20M, is optimized using a rotatable central composite exptl.

design. Two **chromatog.** response functions, based on relative retention and the number of peaks resolved are proposed. Temperature and pressure

gradients giving the best response in the exptl. region are obtained and the effect of their variation on separation is evaluated. Relative standard deviations resulting from both absolute and normalized peak areas are also given.

ST fat soluble vitamin sepn **supercrit chromatog**

IT Vitamins

RL: ANT (Analyte); ANST (Analytical study)

(fat-soluble; optimization of fat-soluble vitamin separation by **supercrit** . fluid **chromatog.**)

IT **Chromatography, supercritical fluid**

Pharmaceutical analysis

(optimization of fat-soluble vitamin separation by **supercrit.** fluid **chromatog.**)

IT 50-14-6, Vitamin D2 58-27-5, Vitamin K3

58-95-7, Vitamin E acetate 59-02-9 67-97-0, Vitamin

D3 68-26-8, Vitamin A 127-47-9, Retinol acetate 11104-38-4, Vitamin K1

RL: ANT (Analyte); ANST (Analytical study)

(optimization of fat-soluble **vitamin** separation by **supercrit** . fluid **chromatog.**)

IT 50-14-6, Vitamin D2 67-97-0,

Vitamin D3

RL: ANT (Analyte); ANST (Analytical study)

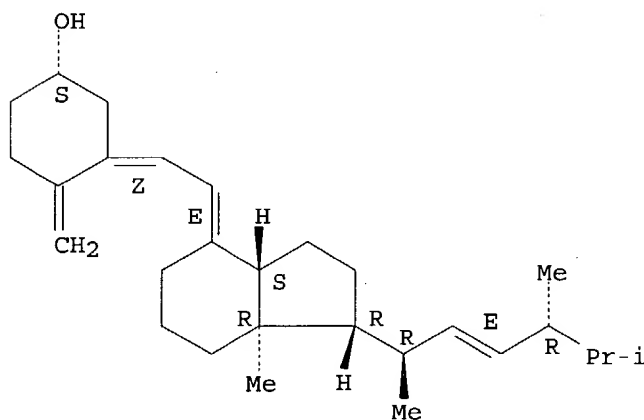
(optimization of fat-soluble **vitamin** separation by **supercrit** . fluid **chromatog.**)

RN 50-14-6 HCAPLUS

CN 9,10-Secoergosta-5,7,10(19),22-tetraen-3-ol, (3 β ,5Z,7E,22E)- (9CI)
(CA INDEX NAME)

Absolute stereochemistry. Rotation (+).

Double bond geometry as shown.

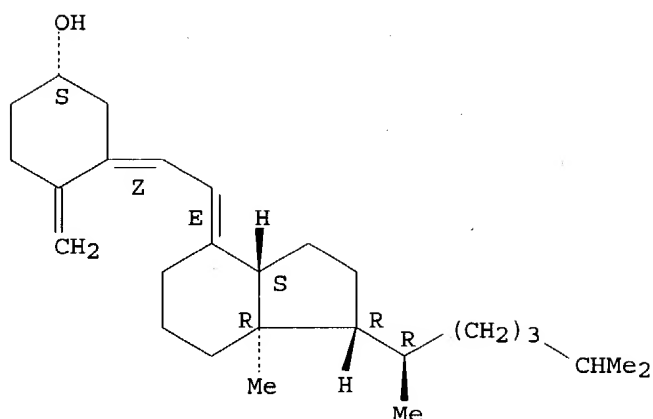


RN 67-97-0 HCAPLUS

CN 9,10-Secocholesta-5,7,10(19)-trien-3-ol, (3 β ,5Z,7E)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.

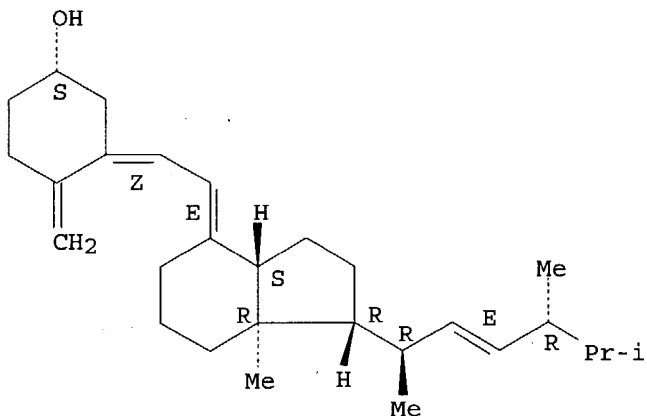
Double bond geometry as shown.



L96 ANSWER 6 OF 9 HCAPLUS COPYRIGHT 2004 ACS on STN
 AN 1988:479795 HCAPLUS
 DN 109:79795
 ED Entered STN: 02 Sep 1988
 TI Analysis of pharmaceuticals and other solutes of biochemical importance by
supercritical fluid chromatography
 AU White, C. M.; Gere, D. R.; Boyer, D.; Pacholec, F.; Wong, L. K.
 CS SFC Res. Cent., Suprex Corp., Pittsburgh, PA, 15238, USA
 SO HRC & CC, Journal of High Resolution Chromatography and Chromatography
 Communications (1988), 11(1), 94-8
 CODEN: HCJCDB; ISSN: 0344-7138
 DT Journal
 LA English
 CC 64-2 (Pharmaceutical Analysis)
 Section cross-reference(s): 9
 AB The feasibility of using **supercrit. fluid chromatog.**
 (SFC) for anal. of polar and/or ionic analytes of interest to the
 pharmaceutical industry is described. Specifically, the analyses of
 cyclosporin (a cyclic undecapeptide), several ionophores, and a group of
 fat-soluble vitamins are illustrated. The separation of a group of fat-soluble
 vitamins was evaluated on 2 bonded stationary phases, DB-5 and DB-WAX
 (Carbowax 20M type, 'bonded'). A new restrictor technol. known as a
 converging-diverging restrictor is described.
 ST gas **chromatog** pharmaceutical analysis; **supercrit** fluid
chromatog pharmaceutical analysis
 IT Ionophores
 (determination of, by **supercrit. fluid chromatog.**)
 IT Pharmaceutical analysis
 (**supercrit. fluid chromatog.** in)
 IT Vitamins
 RL: ANT (Analyte); ANST (Analytical study)
 (fat-soluble, determination of, by **supercrit. fluid chromatog**
 .)
 IT **Chromatography, gas**
 (**supercrit.**, in pharmaceutical anal.)
 IT 50-14-6, Vitamin D2 57-87-4, Ergosterol
 58-27-5, Vitamin K3 58-95-7, Vitamin E acetate 67-97-0,
 Vitamin D3 68-26-8, Vitamin A 84-80-0, Vitamin K1
 127-47-9, Vitamin A acetate 10191-41-0 59865-13-3, Cyclosporin
 92883-58-4 115825-69-9
 RL: ANT (Analyte); ANST (Analytical study)
 (determination of, by **supercrit. fluid chromatog.**)
 IT 50-14-6, Vitamin D2 67-97-0,
 Vitamin D3

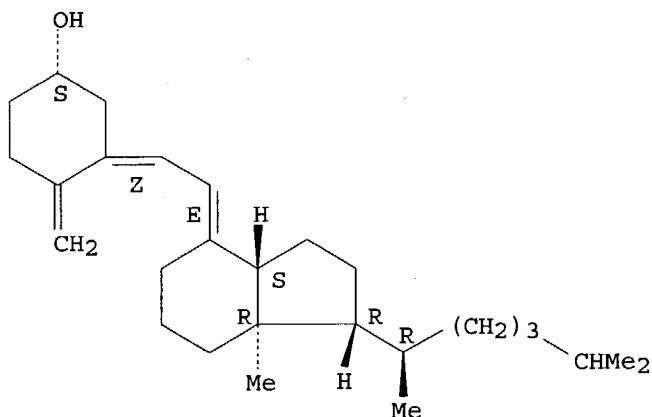
RN 50-14-6 HCAPLUS

Absolute stereochemistry. Rotation (+).
Double bond geometry as shown.



CN 9,10-Secocholesta-5,7,10(19)-trien-3-ol, (3 β ,5Z,7E)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.
Double bond geometry as shown.



AN 1987:81180 HCAPLUS

DN 106:81180

ED Entered STN: 21 Mar 1987

TI Fundamental conditions in pressure-programmed **supercritical**
fluid **chromatography**-mass spectrometry and some applications to
vitamin analysis

AU Matsumoto, K.; Tsuge, S.; Hirata, Y.

CS Fac. Eng., Nagoya Univ., Nagoya, 464, Japan

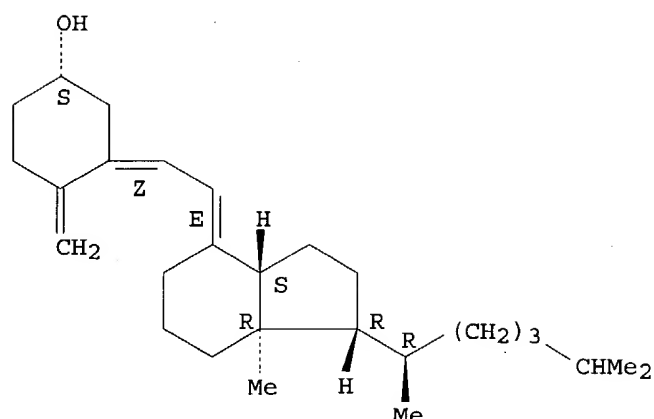
SO Chromatographia (1986), 21(11), 617-21

CODEN: CHRGB7; ISSN: 0009-5893

DT Journal

- LA English
CC 9-15 (Biochemical Methods)
Section cross-reference(s): 80
AB A combined system of pressure-programmed packed capillary **supercrit. fluid chromatog.-mass spectrometry (SFC/MS)** was constructed by using a self-spouting interface assisted by vacuum nebulizing. For the optimum operation of the SFC/MS system, fundamental anal. conditions such as the flow rate of the mobile phase, the pump pressure, and the composition of the mobile phase were examined The use of large packing materials indicated that the capacity factor for a sample solute is almost constant under a given pump pressure regardless of the flow rate of the **supercrit. fluid**. The SFC/MS system was applied to the anal. of both water- and fat-soluble vitamins. Both types of vitamins were clearly separated under basically the same SFC conditions. High quality mass spectra of the vitamins were obtained; selected ion monitoring traces of the vitamins are also reported as well as their UV traces.
ST **supercrit fluid chromatog** mass spectrometry; pressure programmed **supercrit fluid chromatog**; vacuum nebulizing interface **chromatog** spectrometry; vitamin **supercrit chromatog** mass spectrometry
IT Mass spectra
(of fat- and water-soluble vitamins)
IT Mass spectroscopy
(pressure-programmed **supercrit. fluid chromatog.** combined with, of vitamins, fundamental conditions in)
IT Vitamins
RL: ANST (Analytical study)
(separation and identification of, pressure-programmed **supercrit. fluid chromatog.-mass spectrometric**)
IT **Chromatography, gas**
(**supercrit.**, mass spectrometry combined with pressure-programmed, of vitamins, fundamental conditions in)
IT 50-81-7, Vitamin C, analysis 59-67-6, Nicotinic acid, analysis 67-97-0, Vitamin D3 98-92-0, Nicotinamide 8059-24-3, Vitamin B6 11103-57-4, Vitamin A
RL: ANST (Analytical study)
(separation and identification of, pressure-programmed **supercrit. fluid chromatog.-mass spectrometric**)
IT 67-97-0, Vitamin D3
RL: ANST (Analytical study)
(separation and identification of, pressure-programmed **supercrit. fluid chromatog.-mass spectrometric**)
RN 67-97-0 HCAPLUS
CN 9,10-Secocholesta-5,7,10(19)-trien-3-ol, (3 β ,5Z,7E) - (9CI) (CA INDEX NAME)

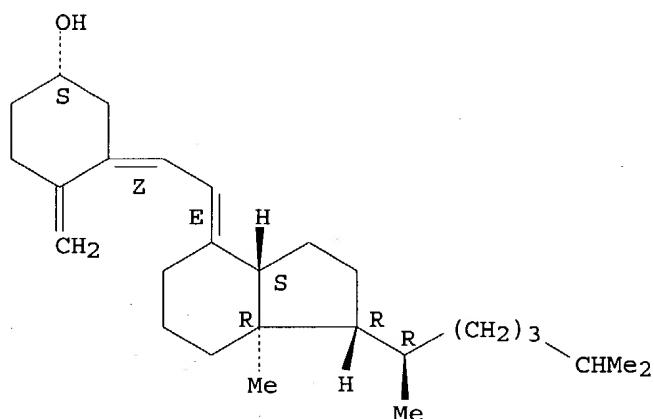
Absolute stereochemistry.
Double bond geometry as shown.



L96 ANSWER 8 OF 9 HCAPLUS COPYRIGHT 2004 ACS on STN
 AN 1985:106428 HCAPLUS
 DN 102:106428
 ED Entered STN: 06 Apr 1985
 TI Separation of hydroxylated **vitamin D3** metabolites by
 high-performance liquid **chromatography**
 AU Moysan, J. F.; Berthou, F.; Floch, H. H.
 CS Lab. Biochim., Fac. Med. Brest, Brest, 29200, Fr.
 SO Pathologie Biologie (1984), 32(8), 825-7
 CODEN: PTBIAN; ISSN: 0031-3009
 DT Journal
 LA French
 CC 2-1 (Mammalian Hormones)
 Section cross-reference(s): 9
 AB The hydroxylated metabolites of **vitamin D3** were separated
 by normal-phase HPLC on Spherisorb 5SW-5, Radpak silica-10 μm , and
 Zorbax silica-5 μm columns and by reversed-phase HPLC on Vydac 201 TP
 C18-5 μm , Radpak C18-10 μm , and Ultraspher C18-5 columns. With the
 normal-phase **chromatog.** systems, the order of elution of the
 hydroxylated metabolites was 25-hydroxyvitamin **D3** and
 (E)-25-hydroxyvitamin **D3**, then (24R), 25-dihydroxyvitamin
D3, 25(S), 26-dihydroxyvitamin **D3**, and
 1 α ,25-dihydroxyvitamin **D3**. However, this order of elution
 was variable with the Radpak silica column as a function of the nature of
 the mobile phase. The level of MeOH in the mobile phase had a
critical effect on the separation and caused the appearance of perfectly
 sym. peaks. With the reversed-phase **chromatog.** systems, the
 order of elution of the metabolites was the opposite of the above elution
 order. In addition, the Z and E isomers of 25-hydroxyvitamin **D3**
 could be separated, in contrast to adsorption **chromatog.**
 ST **vitamin D3** metabolite HPLC; hydroxyvitamin **D3**
 metabolite HPLC; **chromatog vitamin D3**
 metabolite
 IT **Chromatography**, column and liquid
 (high-performance, **vitamin D3** hydroxylated
 metabolites separation by)
 IT **Chromatography**, column and liquid
 (high-performance, reversed-phase, **vitamin D3**
 hydroxylated metabolites separation by)
 IT 67-97-0D, hydroxylated metabolites 19356-17-3
 22350-41-0 32222-06-3 42737-59-7
 55721-11-4
 RL: PROC (Process)
 (separation of, by HPLC)

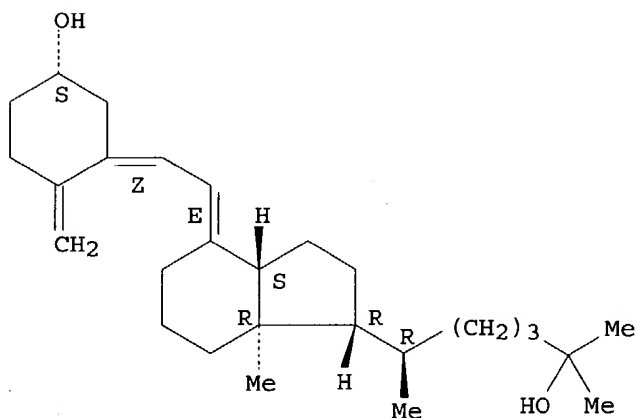
IT 7631-86-9, biological studies 94699-20-4 94699-31-7 94699-49-7
 94699-60-2
 RL: BIOL (Biological study)
 (vitamin D3 hydroxylated metabolite separation by HPLC
 on)
 IT 67-97-0D, hydroxylated metabolites 19356-17-3
 22350-41-0 32222-06-3 42737-59-7
 55721-11-4
 RL: PROC (Process)
 (separation of, by HPLC)
 RN 67-97-0 HCAPLUS
 CN 9,10-Secocholesta-5,7,10(19)-trien-3-ol, (3 β ,5Z,7E)- (9CI) (CA INDEX
 NAME)

Absolute stereochemistry.
 Double bond geometry as shown.



RN 19356-17-3 HCAPLUS
 CN 9,10-Secocholesta-5,7,10(19)-triene-3,25-diol, (3 β ,5Z,7E)- (9CI) (CA
 INDEX NAME)

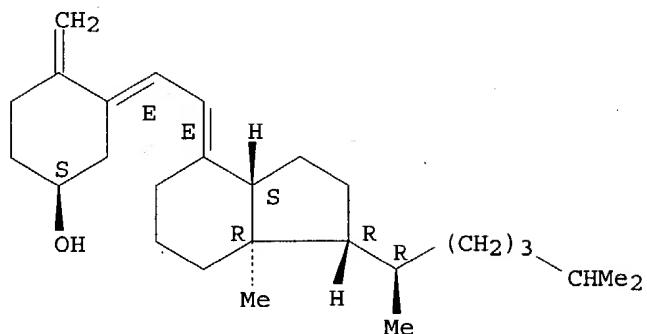
Absolute stereochemistry.
 Double bond geometry as shown.



RN 22350-41-0 HCAPLUS
 CN 9,10-Secocholesta-5,7,10(19)-trien-3-ol, (3 β ,5E,7E)- (9CI) (CA INDEX
 NAME)

Absolute stereochemistry.

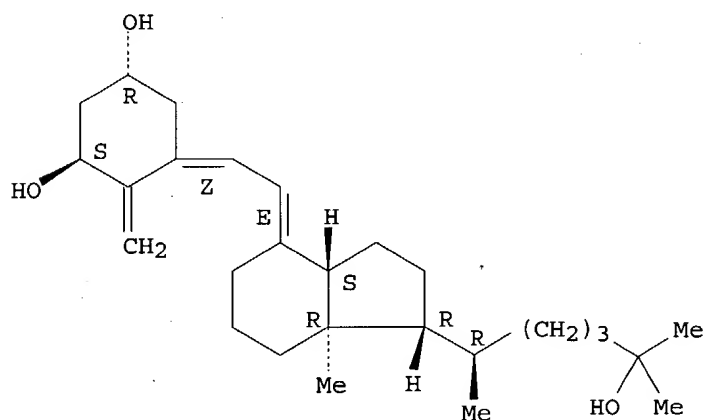
Double bond geometry as shown.



RN 32222-06-3 HCAPLUS

CN 9,10-Secocholesta-5,7,10(19)-triene-1,3,25-triol, (1 α ,3 β ,5Z,7E)-
(9CI) (CA INDEX NAME)

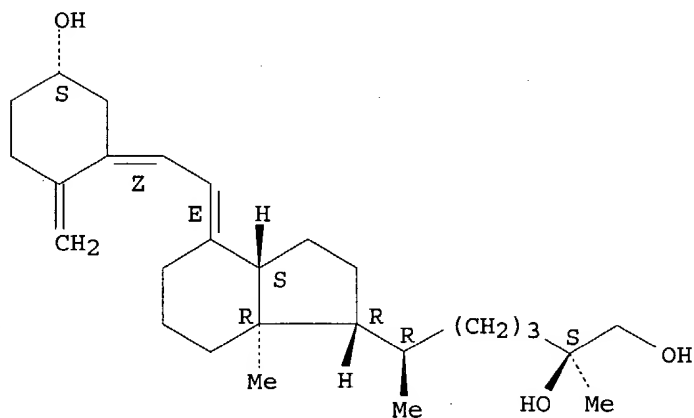
Absolute stereochemistry. Rotation (+).
Double bond geometry as shown.



RN 42737-59-7 HCAPLUS

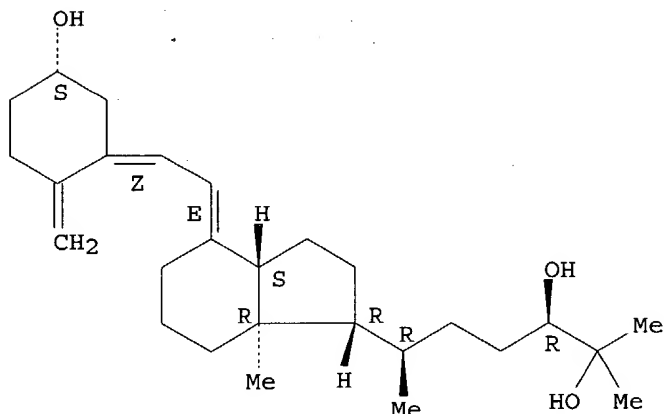
CN 9,10-Secocholesta-5,7,10(19)-triene-3,25,26-triol, (3 β ,5Z,7E,25S)-
(9CI) (CA INDEX NAME)

Absolute stereochemistry.
Double bond geometry as shown.



RN 55721-11-4 HCAPLUS
 CN 9,10-Secocholesta-5,7,10(19)-triene-3,24,25-triol, (3 β ,5Z,7E,24R)-
 (9CI) (CA INDEX NAME)

Absolute stereochemistry. Rotation (+).
 Double bond geometry as shown.



L96 ANSWER 9 OF 9 HCAPLUS COPYRIGHT 2004 ACS on STN
 AN 1967:481297 HCAPLUS
 DN 67:81297
 ED Entered STN: 12 May 1984
 TI Determination of **vitamin D** and of ergosterol in fodder yeasts. I. **Critical** investigation of the test methods in model solutions
 AU Spanyol, Pal; Blazovich, Marta; Gabor, Mrs. Istvan
 CS Kozponti Elemiszertani Kutatóint., Budapest, Hung.
 SO Elelmiszervizsgálati Közlemények (1967), 13(2), 77-91
 CODEN: EMKZAH; ISSN: 0422-9576
 DT Journal
 LA Hungarian
 CC 17 (Foods)
 AB A com. ergosterol (I) preparation was used to test estimation procedures. Good results were obtained by the Liebermann-Burchard color reaction of I with Ac₂O and H₂SO₄, as well as by determining the uv absorption of the complex of I and digitonin. Procedures for **vitamin D** (II) were tested on a com. II preparation. The uv absorption of II itself as well as color reactions with SbCl₃ and with furfural were suitable. In the presence of each other, I and II can be determined by thin-layer chromatog. on silica gel G, using 1:1 cyclohexane:ether and developing with SbCl₃. 63 references.
 ST **VITAMIN D** DETN YEAST; **ERGOSTEROL** DETN YEAST; YEAST
VITAMINS DETN; FODDER YEAST **VITAMINS** DETN; SPANYAR P; BLAZOVICH M; GABOR I MRS
 IT 57-87-4 1406-16-2, **Vitamin D**
 RL: ANT (Analyte); ANST (Analytical study)
 (determination of)
 IT 1406-16-2, **Vitamin D**
 RL: ANT (Analyte); ANST (Analytical study)
 (determination of)
 RN 1406-16-2 HCAPLUS
 CN Vitamin D (8CI, 9CI) (CA INDEX NAME)

*** STRUCTURE DIAGRAM IS NOT AVAILABLE ***

=> d his

(FILE 'HOME' ENTERED AT 14:46:34 ON 08 MAY 2004)
SET COST OFF

FILE 'REGISTRY' ENTERED AT 14:46:51 ON 08 MAY 2004

E VITAMIN D3/CN
L1 1 S E3
E PREVITAMIN D3/CN
L2 1 S E3
E VITAMIN D/CN
L3 1 S E3
L4 STR
L5 50 S L4
L6 8414 S L4 FUL
SAV TEMP L6 QAZI335/A
L7 STR L4
L8 0 S L7
L9 662 S L7 FUL
SAV L9 TEMP QAZI335A/A
L10 9076 S L6,L9
L11 9074 S L10 NOT L1,L2
L12 366 S L11 AND ?VITAMIN?/CNS
L13 8708 S L11 NOT L12
L14 1 S CARBON DIOXIDE/CN

FILE 'HCAPLUS' ENTERED AT 14:52:16 ON 08 MAY 2004

L15 5267 S L1
L16 235 S L2
L17 12688 S (VIT OR VITAMIN) (L)D3
L18 136 S (VIT OR VITAMIN) (L)D 3
L19 2247 S CHOLECALCIFEROL#
L20 14 S COLECALCIFEROL#
L21 249 S (PREVIT OR PREVITAMIN OR PRE() (VIT OR VITAMIN)) (L)D3
L22 6 S (PREVIT OR PREVITAMIN OR PRE() (VIT OR VITAMIN)) (L)D 3
L23 83 S PRECALCIFEROL# OR PRE CALCIFEROL#
L24 125 S PREVITAMIN D
L25 10602 S L3
L26 29557 S VITAMIN D#
L27 16535 S L12
L28 3123 S L13
L29 36305 S L15-L28
E JOHANNSEN M/AU
L30 17 S E3,E8
L31 2 S L30 AND L29
L32 270 S L29 AND (LAROCHE? OR LA ROCHE? OR HOFFMANN?)/PA,CS
L33 1 S US20010001801/PN OR EP98-111490/AP,PRN
L34 176984 S L14
L35 441586 S CARBON DIOXIDE OR CO2
L36 204 S CARBONDIOXIDE
L37 184 S L29 AND L34-L36
L38 4 S L37 AND L31,L32,L33
E CHROMATOGRAPHY/CT
L39 1 S L37 AND E30
L40 3 S L37 AND E110
L41 2 S L37 AND E145,E151,E154
L42 0 S L37 AND E158
L43 2 S L37 AND E27-E29
E E3+ALL
L44 2 S L37 AND E4-E6
L45 12 S L37 AND E3+NT
E SILICA GEL/CT

L46 2 S L37 AND E4-E28
 E E3+ALL
 L47 2 S L37 AND E16,E15+NT
 L48 18 S L37 AND (?SUPERCRIT? OR ?SUPER CRIT?)
 L49 1 S L37 AND FLASH?
 L50 23 S L33,L38-L49
 E SEPARATION/CT
 L51 561 S E3+OLD,NT,PFT AND L29
 E PURIFICATION/CT
 L52 9 S E3+OLD,NT,PFT AND L29
 E ISOLATION/CT
 L53 561 S L51-L52
 L54 19 S L37 AND (?SUPERCRIT? OR ?SUPER CRIT? OR FLASH?)
 L55 0 S L37 AND (BACKPRESSUR? OR BACK PRESSUR?)
 L56 22 S L37 AND ?CHROMATOG?
 L57 30 S L54,L56,L50
 L58 20 S L57 AND (PY<=1999 OR PRY<=1999 OR AY<=1999)
 L59 473 S (PROVIT? OR PRO VIT?) ()D#
 L60 172 S L59 NOT L29
 L61 163 S L60 AND (PY<=1999 OR PRY<=1999 OR AY<=1999)
 L62 23 S L61 AND ?CHROMATO?
 L63 0 S L61 AND (BACKPRESSUR? OR BACK PRESSUR?)
 L64 0 S L61 AND (?SUPERCRIT? OR ?SUPER CRIT? OR FLASH?)
 L65 7 S L61 AND L34-L36
 L66 2 S L61 AND CHROMATOGRAPHY+OLD,NT,PFT/CT
 L67 2 S L61 AND SEPARATION+OLD,NT,PFT/CT
 L68 0 S L61 AND PURIFICATION+OLD,NT,PFT/CT
 L69 49 S L62,L65-L67,L58
 L70 49 S L69 AND (PY<=1999 OR PRY<=1999 OR AY<=1999)
 L71 10 S L57 NOT L70
 L72 8 S L70 AND ?CRITIC?
 L73 27 S L70 AND L34-L36
 L74 27 S L72,L73
 L75 22 S L70 NOT L74
 SEL DN AN L74 25
 L76 26 S L74 NOT E1-E3
 L77 36 S L71,L76
 L78 12 S L77 AND (?RADIAT? OR UV OR ULTRAVIOL? OR ULTRA VIOL?)
 L79 36 S L77,L78

FILE 'REGISTRY' ENTERED AT 15:17:03 ON 08 MAY 2004

FILE 'HCAPLUS' ENTERED AT 15:18:23 ON 08 MAY 2004

L80 36477 S L29,L59
 L81 471 S L80 AND ?CRITIC?
 L82 28 S L80 AND ?SUPERCRITIC?
 L83 0 S L80 AND ?SUPER CRITIC?
 L84 0 S L80 AND ?ULTRACRITIC?
 L85 36 S L81 AND ?CHROMATOG?
 L86 44 S L82,L85
 L87 21 S L81 AND L34-L36
 L88 29 S L86,L87 NOT L79
 SEL DN AN 6 9-12 14-17 19 22 23 24 26
 L89 14 S L88 AND E4-E45
 L90 2 S L89 AND SILICA GEL
 L91 14 S L89,L89
 L92 7 S L91 AND L82
 L93 8 S L90,L92
 L94 6 S L91 NOT L93
 SEL DN AN 2
 L95 1 S E46-E48 AND L94
 L96 9 S L93,L95

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